

CHEMICAL ABSTRACTS

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I—APPARATUS

C. G. DERICK

Sulfuric acid concentrator and vacuum pump. O. MAASS. *J. Am. Chem. Soc.* **42**, 2871-4(1920).—This pump in which H_2SO_4 is the confining liquid is designed for vacuum distn. and is particularly adapted to the special case where the liquid to be distd. is an aq. soln. which is to be freed partly or wholly from its water content. The pump has given excellent results in the fractional distn. (without cracking) of oil extd. from tar sands, and in the concn. of dil. H_2O_2 solns.

JAMES M. BELL

Preparation of pure carbon dioxide. R. C. FARMER. *J. Chem. Soc.* **117**, 1448-7 (1920).—A continuous automatic generator for pure CO_2 is described and a sketch of the app. given. Solns. of KHCO_3 , 300 g. per l. and H_2SO_4 , 120 cc. per l. are mixed, each being freed from air by bubbling CO_2 through it before mixing. The generator has proved useful in testing the *stability of gun cotton* by Will's method (*Z. angew. Chem.* **14**, 743, 774(1901)).

A. R. M.

A new conductivity cell. FRANK E. RICE. *J. Ind. Eng. Chem.* **12**, 1202(1920).—In this cell the electrodes are Pt bands which fit snugly around a glass tube and are sealed to Pt wires which are blown through the glass and extend out one end of the tube. The tube is closed at both ends with rubber stoppers through which a thermometer extends. Measurements are made by holding the tube upright in the soln. The distance between the electrode bands and their size is gaged by the cond. of the soln. to be measured. The cell is easily made and cleaned but not of great accuracy.

G. W. STRATTON

An improvement in the Haldane general air analysis apparatus. ROBT. C. FREDERICK. *Analyst* **45**, 409-10(1920).—The improvement consists of using a combustion chamber in which the wide part is $\frac{2}{8}$ in. shorter in length, the stem $1\frac{1}{8}$ in. longer, and the mark on the stem is $\frac{1}{2}$ in. from the shoulder ($\frac{2}{8}$ in. higher). These changes do not necessitate any rearrangement of the other parts.

F. W. SMITHER

Apparatus for grinding rock specimens. A. HADDING. *Geol. För. Förh.* **42**, 378-83(1920).—An app. tested at Lunds Geologisk-Mineralogiska institution filled these requirements satisfactorily: several specimens of varying thickness could be prepd. at the same time, each specimen finished could be easily removed and another put in its place without stopping the grinding of the other specimens, the grinding of each specimen was automatically stopped when the specimen reached a predetd. thickness, the app. could be left running without attention, and it was moderately simple and inexpensive. Two cuts and 1 diagram explain its construction clearly. The rock specimens are fastened with wax to the bottoms of 8 rock holders (Fe disks with shafts) held in the periphery of a circular metal plate properly supported above a rotating cast-iron plate onto which water and carborundum powder are fed. Springs hold the specimens, both thick and thin, against the plate with equal pressure. A special device prevents individual specimens from being completely ground away although the app. is still running. The rotating plate may be driven by an electric motor or by hand or foot power. It is recommended that the app. be used for rough grinding and that the final fine grinding be done by hand.

WILHELM SEGERBLOM

The Priest photometer. G. W. AGE. *Cotton Oil Press* 4, No. 9, 42-3(1921).—The instrument designed by Priest for the color grading of cotton seed and other oils (*C. A.* 14, 133) has many advantages over the Lovibond glasses. With it the light transmitted by an oil is compared directly with that transmitted by a known transmission standard. The transmittance is measured for 3 wave lengths, red, yellow, and green and not color but the brightness in each of these spectrum divisions is compared. A color-blind person can make reading without difficulty. Observations with the photometer express characteristics of the oil and are not dependent upon properties inherent in the instrument or the observer's eye. While these observations can be translated into terms of the present Lovibond glasses, ultimately a different system of reporting the readings would probably be adopted.

H. S. BAILEY

Standardizing mercury thermometers. SIEBERT AND KARL SCHEEL. *Z. Angew. Chem.* 33, I, 216(1920).—Greater uniformity in diam. and length of thermometers is recommended, and desirable dimensions are given.

W. P. WHITE

Electric oven for rapid moisture tests. GUILFORD L. SPENCER. *J. Ind. Eng. Chem.* 13, 70-2(1921).—The oven, which was devised primarily for detg. moisture in raw sugars, is described in detail (U. S. pat. 1,348,757).

F. W. SMITHER

A new illuminator for microscopes. III. ALEXANDER SILVERMAN. *J. Ind. Eng. Chem.* 12, 1200-2(1920).—Changes in construction of the lamp, rheostat and stage adapter and suggested applications of the illuminator previously described (*C. A.* 11, 2977; 12, 1606) are given.

G. W. STRATTON

Effect of burners and bars on hot-plate efficiency. F. S. LARKIN. *Gas World* 73, 465-6(1920); *Gas J.* 152, 520-4(1920).—For general purposes a burner should be capable of passing sufficient gas to ensure rapid boiling with economy; when turned low the heat should be spread over as large a surface as possible to prevent the burning of thick liquids; and it should be "elastic" that is of a type which will not smother badly with change of gas pressure. The best angle of drilling for the gas flame is 30° from the vertical outside and 45° inside. This distributes the gas most satisfactorily and gives ample chance for secondary air to mix with the flame. Bars lower the heat efficiency of a burner; in one case to as much as 35%. The distance of the bars apart affect the result to some extent, but the 3 most important factors are ventilation, wt. of metal and depth of bar. Tabulated data from several tests with various types of burners are given.

J. L. WILEY

The Sugg-Larkin hot plate. ANON. *Gas World* 73, 467-8(1920); *Gas J.* 152, 636-7(1920); cf. preceding abstr.—This plate retains both the features of a solid hot plate for multiple boiling and the open flame for boiling a single vessel. Its efficiency in raising a qt. of water from 54° to h. p. as compared with 2 standard types of burners was 4 min. 47 sec. with a consumption of 1.84 cu. ft. of gas. An av. saving of 107 cu. ft. of gas a week in ordinary cooking is possible.

J. L. WILEY

The ultramicrometer (WHIDDINGTON) 3. Centrifugal extractors and separators applied to the chemical industry (BROADBENT) 13.

METZ, CARL AND HAGER, HERMANN: *Das Mikroskop und seine Anwendung. Handbuch der praktischen Mikroskopie und Anleitung zu mikroskopischen Untersuchung.* 12th Ed. revized. Berlin: Julius Springer. M. 33. For review see *Chem.-Ztg.* 44, 855(1920).

Acetylene generator. H. F. PRILLWITZ. U. S. 1,360,682, Nov. 30.

Filter for water or other liquids. J. P. PROBST. U. S. 1,361,493, Dec. 7.

Apparatus for the treatment of one liquid with another. A. E. HOLLEY and O. E. MORR. Can. 206,338, Dec. 7, 1920. The app. comprizes a series of agitators all

at the same level, a series of separators at the same level as the agitators, the bottom of a separator being connected to the bottom of an agitator in one direction and the top of a separator to the top of an agitator in the other direction and each separator being in direct communication with the agitator at a point situated between the other connections.

Apparatus for heating, evaporating or calcining materials. JNO. S. MORGAN. Can. 206,648, Dec. 21, 1920. App. is specified for heating, drying or carbonizing by immersing a material in a bath of inert molten metal and collecting the gases.

Spraying apparatus. AARON S. NICHOLS. Can. 206,656, Dec. 21, 1920. A spraying app. has rotatable disks placed side by side at inclinations to their axis and arranged to dip into a body of liquid.

Apparatus for purifying gas. HARRY F. SMITH. Can. 206,371, Dec. 7, 1920.

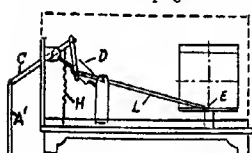
Air washers. KIRKE K. WYATT. Can. 206,578, Dec. 14, 1920.

Removing gases from vacuum, etc., apparatus. G.-HOLST, E. OOSTERHUIS and NAAMLooZE VENNOOTSCHAP PHILIPS' GLOEILAMPENFABRIEKEN. Brit. 151,611, Sept. 22, 1920. In vacuum, etc., app. such as thermionic valves, Röntgen-ray tubes, or incandescent lamps, gaseous residues are removed or inert gases purified by means of an alloy of an alkali or alkali-earth metal, such as Ca, Ba, Sr, K or Na, with a nobler metal having a low vapor pressure, such as Sn, the alloy being placed in the bulb so as to be heated in order to vaporize the alkali, etc., metal, while the other metal does not vaporize perceptibly. The nobler metal protects the alkali, etc., metal against moisture and oxidation. An alloy with Sn may be applied as a solder upon a metal part in the bulb; an alloy having a higher m. p. may be used as material for one or more electrodes or conductors.

Filling material for reaction towers. C. J. GOODWIN. U. S. 1,360,928, Nov. 30. A filling material for reaction, concg. or absorbing towers is formed in the shape of 2 hollow truncated cones joined at their smaller ends and open from end to end.

Specific-gravity tester. J. H. KESSLER. U. S. 1,360,537, Nov. 30. The device is adapted for testing the sp. gr. of fruit juices during cooking. It comprizes a pair of similar bulbs connected by a narrow neck, one bulb being provided with a narrow graduated stem and the other with a stopcock with an offset outlet pipe.

Apparatus for estimating specific gravity. F. L. HALLIWELL. Brit. 149,448, Nov. 17, 1919. The pat. relates to app. of the type in which a float or sinker is suspended in the fluid whose sp. gr. is to be measured by a wire, etc. A' attached to a balance



lever C connected to a multiplying lever L carrying a recording pen, etc., E . C is weighted on the side connected to the multiplying lever by an arm D , which is placed at such an angle that, in the zero position, the arm is at a considerable angle to the vertical, in order to enable a more open scale to be employed. The balance lever may also be under the control of a spring H . The float is contained in a totally immersed still-fluid chamber perforated at the top and bottom.

Acetylene gas lamps. M. LEONHARDY. Can. 206,675, Dec. 21, 1920.

Miners' lamps. EDWARD M. ERICKSON. Can. 206,433, Dec. 14, 1920.

2—GENERAL AND PHYSICAL CHEMISTRY

WILLIAM E. HENDERSON

Augustus George Vernon Harcourt. H. B. D. *J. Chem. Soc.* 117, 1626-31 (1920).—An obituary. E. J. C.

Lucius Trant O'Shea. W. C. W. AND H. L. *J. Chem. Soc.* **117**, 1631-3(1920).—An obituary. E. J. C.

James Emerson Reynolds. T. E. THORPE. *J. Chem. Soc.* **117**, 1633-7(1920).—An obituary. E. J. C.

Watson Smith. P. P. B. *J. Chem. Soc.* **117**, 1637-8(1920).—An obituary. E. J. C.

Alfred Werner. G. T. MORGAN. *J. Chem. Soc.* **117**, 1639-48(1920).—An obituary. E. J. C.

The properties of subsidiary valence groups. I. The molecular volume relationships of the hydrates and amines of some cobalt compounds. GEORGE L. CLARK, A. J. QUICK AND WM. D. HARKINS. Univ. Chicago and Vanderbilt. *J. Am. Chem. Soc.* **42**, 2483-96(1920).—The first of a series of papers dealing with the effect of vols. of cation, anion and subsidiary valence groups, and of interatomic and intermol. electromagnetic fields upon stability of compds. in which subsidiary valence is displayed by the central atom. A study is made of how the groups change in vol. as they are packed into the *space lattice cavities* of the crystal, to indicate the vol. of the cavities, the limiting compressibility of the group and its consequent apparent activation. The compds. used are the hydrates and amines of CoCl_2 and CoSO_4 . The methods of prepn. of 2 hydrates (6 and 2 H_2O), 4 amines (6, 5 (new), 4 and 2 α and β NH_3), 1 hydrated ammine (5 $\text{NH}_3\cdot\text{H}_2\text{O}$, new) of CoCl_2 , and 3 hydrates (7, 6 and 4 H_2O), 1 ammine (5 NH_3) and 1 hydrated ammine (4 $\text{NH}_3\cdot 2\text{H}_2\text{O}$, new) of CoSO_4 are given. From the very accurately detd. sp. gr. of each, the mol. vol., apparent vol. of the subsidiary group and % compression of the group over its free vol. are calcd. H_2O shows an av. compression of 20%, and NH_3 of 30% as compared with 40% in 3 reference cobaltic chloride amines. Announcement is made of the prepn. of *cobaltous decammine chloride*, light brown in color, sp. gr. 1.71, the formation of which is governed by very special conditions. II. Subsidiary group mobility as studied by the heat decomposition of some cobaltamines. *Ibid* 2496-8.—The mechanism of the heat decompn. of cobaltous and cobaltic amines is compared. The high compression of NH_3 and the easily reducible Co atom in the latter amines result in a disruption of the mol. according to the following equations for the 3 salts used: $6\text{CoCl}_2\cdot 6\text{NH}_3 \longrightarrow 6\text{CoCl}_2 + 6\text{NH}_4\text{Cl} + \text{N}_2 + 28\text{NH}_3$; $2\text{Co}[(\text{NH}_3)_5(\text{NO}_2)]\text{Cl}_2 \longrightarrow \text{Co}_2\text{O}_3 + 5\text{NH}_3 + 4\text{NH}_4\text{Cl} + 2\text{N}_2 + \text{H}_2\text{O}$; and $2\text{Co}(\text{NH}_3)_5(\text{NO}_3)_2 \longrightarrow 6\text{N}_2 + 9\text{H}_2\text{O} + \text{Co}_2\text{O}_3$.

Active or available chlorine. J. R. MACMILLAN. *Chem. Met. Eng.* **23**, 1064 (1920).—The purpose of this note is to correct 2 fallacious views: (1) that when Cl_2 is absorbed in an alk. soln. only $\frac{1}{2}$ of it can be active because the other half forms chlorides, whereas the active principle is really O, one atom of which is replaced in $\text{Ca}(\text{OCl})_2$ by 2 atoms of Cl; and (2) that since the percentages of active and total Cl as detd. are the same while only $\frac{1}{2}$ is in hypochlorite, an efficiency of 200% is attained, whereas this is made possible because the oxidizing power of the soln. is really what is detd. although expressed in terms of Cl. G. L. CLARK

The chemistry and crystallography of some fluorides of cobalt, nickel, manganese and copper. FLOYD H. EDMISTER AND HERMON C. COOPER. Syracuse Univ. *J. Am. Chem. Soc.* **42**, 2419-34(1920).—Crystals of fluorides of the compn. $\text{RF}_2\cdot 5\text{HF}\cdot 6\text{H}_2\text{O}$, where R is Co, Ni, Mn, or Cu, were prepd. Crystallographic measurements showed that the Co, Ni and Mn compds. are rhombohedral and apparently isomorphous; the Cu salt is monoclinic. The compd. $\text{CuF}_2\cdot 2\text{H}_2\text{O}$ was obtained as small crystals, too imperfect for measurement. RALPH W. G. WYCKOFF

The properties of pure hydrogen peroxide. I. O. MAASS AND W. H. HATCHER. *J. Am. Chem. Soc.* **42**, 2548-69(1920).—Pure H_2O_2 was prepd. from ordinary 3% aq. soln. in four stages: (1) concn. to 30% soln. by the sulfuric acid concentrator (cf. C. A. **5**, 453); (2) distn. to remove non-volatile impurities; (3) concn. of the 30% pure soln.

to 90%; and (4) sepn. of pure H_2O_2 by fractional crystn. A yield of 50% of pure hydrogen peroxide was obtained. The freezing point is -1.70° and greater supercooling than with water is possible (as low as -30°). The density of liquid hydrogen peroxide is 1.4419 at 19.90° , 1.4631 at 0.1° and 1.4774 at -12.13° , the av. decrease in d. per degree rise being 0.001075. The d. of H_2O - H_2O_2 mixts. at 0° and 18° varies nearly linearly. The density of solid hydrogen peroxide is 1.6434 at -4.45° , the contraction on freezing being about 11%. The viscosity of hydrogen peroxide is slightly greater at 0° than that of water (0.01828 and 0.1778), while at higher temps. the difference becomes greater. For H_2O - H_2O_2 mixts. there is at 0° a marked minimum at 15% H_2O_2 and a max. at 70% H_2O_2 . At 18° the max. and minimum degenerate into discontinuities. The surface tension of hydrogen peroxide is 78.73 dynes at 0.2° and 75.94 dynes at 18.2° . From Ramsay and Shields's formula these values indicate an association of 3.48. The value for water is 3.58. For aq. solns. the curve of surface tension-compn. is a straight line. The specific heat of liquid hydrogen peroxide is 0.579 and of solid hydrogen peroxide is 0.470; the latent heat of fusion is 74 cal., all with a probable error of 4%. The refractive index at 22° is 1.4139 and from the Lorenz-Lorentz formula the molecular refractivity is 5.900. This value is higher than the value calcd. for $\text{HO}-\text{OH}$ (5.33) and is much lower than that for $\text{HO}\equiv\text{OH}$. Contrary to a prevailing impression, ether and H_2O_2 are but little sol. in one another. H_2O_2 dissolves neutral salts readily and at higher temps. acts on glass, so that the detn. of the h. p. is not practicable in glass vessels. Contact with most metals causes decompn. Living tissue is not injured by it, although the skin becomes absolutely white at first, changing to normal again after a few hours. Dead tissue is destroyed and removed by H_2O_2 . Pure aq. solns. in a suitable container keep indefinitely. Decompn. is caused by foreign substances and seems to be less in the stronger solns.

JAMES M. BELL

The properties of pure hydrogen peroxide. II. O. MAASS AND O. W. HERZBERG. *J. Am. Chem. Soc.* 42, 2569-70(1920).—This paper deals solely with the freezing-point curve of solns. of H_2O_2 and H_2O . There are three branches, the solids phases being H_2O (ice), $\text{H}_2\text{O}_2 \cdot 2\text{H}_2\text{O}$ (m. p. -51°) and H_2O_2 , with eutectic points at -52° and 45° H_2O_2 and at -56° and 59° H_2O_2 .

JAMES M. BELL

Dielectric constant of selenium oxychloride. JAMES E. WILDISH. *J. Am. Chem. Soc.* 42, 2607-9(1920).—The dielec. const. is detd. at three temps. The values are 46 ± 1 at 20° , 51.0 ± 0.5 at 10° , and 17 ± 2 at 0° where the substance is solid.

C. R. PARK

The molecular state of water vapor. JAMES KENDALL. *J. Am. Chem. Soc.* 42, 2477-82(1920).—The author's summary is: The monomolecular nature of water vapor has recently been questioned, "spontaneous ionization" of H_2O into H^+ and OH^- at low temps. and association into $(\text{H}_2\text{O})_2$ at higher temps. being postulated. It has been shown here that the evidence for the existence of the equilibria $\text{H}_2\text{O} \rightleftharpoons \text{H}^+ + \text{OH}^-$ and $2\text{H}_2\text{O} \rightleftharpoons (\text{H}_2\text{O})_2$ is in each case entirely insufficient; that the abnormalities in the vapor densities of normal substances at their boiling points are similar to that in water vapor at 100° ; and that such abnormalities practically vanish on applying the corrections for deviation from the perfect gas laws. An "equation of state" correction is similarly necessary in gas reactions at high pressures, in calculations on dissociation in the vapor state, and in Dumas or Victor Meyer vapor-density determinations.

JAMES M. BELL

Derivation of a formula for the temperature dependence of the velocity constants in gas reactions from a special image of the process. J. TRESLING. *Proc. Roy. Acad. Amsterdam* 23, 143-8(1920).—The dissociation of I is considered as an example. The special image of the process assumes that an I atom is a center of force and that it will act on neighboring atoms only when their distance lies between a and $a + da$ where a is the radius of the attraction sphere of the atom. Each pair of the atoms which lie

in their mutual spheres of attraction forms a mol. It is further assumed that there is a thin layer just outside of the attraction layer in which the forces of the atoms are repellent. The theory of quanta is not involved in the considerations of this paper. Three formulas are derived: $\log K = -[(\psi_1 + \psi_2)/kT] + \log(\omega/2)$; $\log k_1 = (\psi_1/kT) + \frac{1}{2} \log(kT/\pi m) + \log(S/\omega)$; $\log k_2 = -(\psi/kT) + \frac{1}{2} \log(kT/\pi m) + \log(S/2)$. In these formulas, K is the equil. const., ψ_1 and ψ_2 are the energies gained by an atom coming from the outside and passing through the two layers (one of attraction and one of repulsion), k is a const., k_1 and k_2 are the reaction velocities, ω is the vol. of the sphere of attraction, and S the surface of the attraction layer. D. MACRAE

The specific heat of saturated vapors and the entropy-temperature diagram of certain fluids. J. A. EWING. *Phil. Mag.* [6] 40, 501-2(1920).—This is an additional note to C. A. 14, 3565. S. C. L.

A series of abnormal Liesegang rings. E. HATSCHKE. *Kolloid.-Z.* 27, 225-9 (1920).—The rings were formed by layering solns. of Ca salts (some mixed, some containing NaCl) over jellies containing 10 g. gelatin in 100 cc. of 2-3% cryst. Na_3PO_4 . Most of the gelatin solns. were prepd. by soaking flake gelatin in the phosphate soln. for 24 hrs., warming to 90° and filtering. This method of prepo. was varied and several kinds of gelatin were used, one tube being hardened by formaldehyde. The anomalies exhibited by the rings are illustrated and described in detail—convexities pointing downward, twin rings, screw-like forms, etc. In one case there appeared a ring of macroscopic crystals. As Liesegang has shown, the kind of gelatin, its acidity and gelatose content, as well as the concn. of the reagents greatly influence ring formation, but these expts. show that rather complicated structures can be formed by simple diffusion. [The process seems to be influenced by the protective action of gelatose, the nature and percentage of which varies according to grade of gelatin and method of prepn. of the jelly.—ABSTRACTOR.] JEROME ALEXANDER

A capillary phenomenon. H. BRECHOLD. *Kolloid.-Z.* 27, 229-33(1920).—On attempting to impregnate porous cells (unglazed porcelain, burned kieselguhr) with an insol. ppt. produced, *i. e.*, by the interaction of AgNO_3 with KCNS or of CuSO_4 with NaOH, it was found that the reaction product was concd. mainly in the exterior layer of the cell wall, although one salt was uniformly distributed initially by soaking the cell in its soln. This concn. is consequent upon the accumulation of the salt in the exterior layer upon drying, which was necessary to insure the penetration of the second reagent. The phenomenon seems to be a general one. Ordinary garden earth when wet with solns. of NaCl, BaCl_2 , and $\text{Al}_2(\text{SO}_4)_3$ and then allowed to dry slowly, showed enormous concn. of the salts in the upper layers. The importance of the phenomenon in agriculture and geology is discussed, and also its relation to efflorescence on brick-work, weathering of saltpeter, etc. The theory is being investigated, the most probable explanation being the growth of larger crystals at the expense of smaller ones. J. 'A.'

Conductivity and viscosity of solutions in dimethylamine, trimethylamine, ethylamine, diethylamine, triethylamine and propylamine. HOWARD McKEE ELSKY. *J. Am. Chem. Soc.* 42, 2454-76(1920).—All are found to be very poor solvents and poor ionizing agents. The max. equiv. cond. was of the order of magnitude of one reciprocal ohm. The densities of the solvents are given to three figures. All measurements were made at -33.5°. C. R. PARK

The ionization of aqueous solutions of ammonia in the presence of urea. WINTHROP M. BURKE. *J. Am. Chem. Soc.* 42, 2500-6(1920).—The ionization const. for NH_4OH in water at 25° is 18.12×10^{-6} , and in urea soln. (2 M) is 12.92×10^{-6} . This result does not accord with the Nernst-Thomson hypothesis that in solvents of higher dielec. const. than water the ionization is in general higher than in water, for the dielec. consts. are 78.83 for water and 83.98 for 2 M soln. of urea at 18°. The result cannot be wholly accounted for by viscosity as the viscosity of this urea soln. is

only about 3% higher than that of water. From the great tendency of urea to form complexes with acids, bases and salts, it is suggested that possibly there was formed an ammonium-urea complex having an ionization const. less than that of NH_4OH .

JAMES M. BELL

Surface tension and molecular attraction. The adhesional work between mercury and organic liquids. WM. D. HARKINS AND E. H. GRAFTON. Univ. Chicago. *J. Am. Chem. Soc.* 42, 2534-8(1920); cf. *C. A.* 14, 3343.—This preliminary paper includes a table of the work of adhesion between Hg and org. liquids, calcd. from the equation $W_A = -\Delta\gamma = \gamma_{\text{Hg}} + \gamma_i - \gamma_{\text{Hg},i}$. The surface tension of pure Hg detd. by the Harkins method is about 464 dynes in air and 374.8 in H_2O . Energy relationships are considered in a paper immediately following.

G. L. CLARK

The surface energy of mercury and the energy relations at the interface between mercury and other liquids. WM. D. HARKINS AND WARREN W. EWING. Univ. Chicago. *J. Am. Chem. Soc.* 42, 2539-47(1920); cf. preceding abstract.—In this paper are considered and tabulated the complete data on energy relations obtained from measurements of the surface tension of Hg at various temps. *in vacuo*, the interfacial surface tension between Hg and various liquids and vapors at 20°, and the interfacial tension between Hg and 4 org. liquids at temps. between 0° and 60°. Methods of measurement which are modifications of those used in previous work with H_2O are fully described. For the results and comparisons of adhesional and cohesive work cf. *C. A.* 14, 3343. In addition it is shown that adhesional work decreases rapidly with increase in temp., while the total adhesional energy, defined by the eq. $E_A = (\gamma_{\text{Hg}} + l_{\text{Hg}}) - (\gamma_i + l_i) = (\gamma_i + l_i)$, where γ is surface energy, l is the latent heat or $T\Delta\gamma/\Delta T$, and the subscripts i and g refer resp. to liquid and interface.

G. L. CLARK

Normal vapor pressure. C. v. RECHENBERG. *J. prakt. Chem.* 101, 112-22 (1920).—The extrapolation of b. ps. of liquids at reduced pressures to other pressures including the normal one of 760 mm. is considered from the standpoint of the use of the Crafts-Young formula. The disagreement in reported values of b. ps. at reduced pressures is to be found partly in inaccuracies of measurement of pressure, since a difference in 1 mm. pressure makes an error of several degrees in temp. while an error of 1° in temp. results of course in a difference of only a small fraction of a mm. A comprehensive table of liquids is compiled from the literature, and includes the b. p. at 760, 14, 15 and 16 mm. pressure, and the coeff. x in the Crafts-Young formula $C = x(15 - p)(273 + t)$, where C is the correction value in temp. degrees to be added or subtracted from the temp. t of the satd. vapor at 15 mm., p is the pressure at which the corresponding boiling temp. is sought (either 14 or 16 mm. here), and x is the coeff., different for different classes of liquids and detd. as the mean of the values of h. p. changes from 14 to 15 mm. and 15 to 16 mm. This coeff. is found to have const. values which may be used without sensible error as follows: 0.004187, hydrocarbons, ethers, oxides, mustard oils, thiophenes, mercaptans, sulfides, nitriles and acid chlorides; 0.004009, ketones; 0.003946, esters, amines and aldehydes; 0.003720, phenols; 0.003574, acids; 0.003458, alcs.; 0.00301, hydrazones; 0.002835, quinones.

G. L. CLARK

Statistical mechanics applied to chemical kinetics. RICHARD C. TOLMAN. *J. Am. Chem. Soc.* 42, 2506-28(1920).—Thermodynamics furnishes the principle that chem. reactions can take place which are accompanied by an increase of entropy. The rate of approach to equil. is still a baffling question, which T. considers from the point of view of statistical mechanics. The ordinary equation for reaction velocity is based on considerations of the number of collisions between reacting mols. A modification of this idea by Arrhenius postulates that chem. reaction occurs only when the colliding mols. are in a special activated state. The energy of activation is supposed by Perrin and others to be due to radiation. Perrin's theoretical treatment is open, however, to numerous criticisms. T. using the results of statistical mechanics considers

the rate of monomol. reactions in dil. gaseous systems, the temp. coeff. of these reactions, the photochem. rate of monomol. reactions and their temp. coeffs., the last being small. This result is confirmed by results on photochem. reactions. There is also a brief consideration of bi- and polymol. reactions from the same view-point. See *J. Am. Chem. Soc.* 43, 126-7(1921) for correction on original paper.

JAMES M. BELL

Possible relation between mechanical, chemical and electrical quantities. CARL HERING. *Science* 52, 509-10(1920).—If the electrochem. equiv. of Ag were 0.3% higher the electrolytic relation grams $\times g = 10 \times$ coulombs \times at. wt./g, where g is the acceleration due to gravity. The faraday becomes $g^2/10$ or 96,157. Certain physical dimensions are not clear but correction for terrestrial factors and intra-atomic accelerations might make the relation exact, and there may well be relations between chem. and elec. quantities which have not yet been brought to light. A. E. STEARN

Study of the saturated potassium chloride calomel cell. HAROLD A. FALES AND WILLIAM A. MUDGE. *J. Am. Chem. Soc.* 42, 2434-53(1920).—Comparative measurements are made by opposing to the electrode H_2 , HCl 0.1 M the following three electrodes: Hg-HgCl, KCl 0.1 M ; Hg-HgCl, KCl 1 M ; and Hg-HgCl, satd. KCl. The e. m. f. of the satd. calomel electrode was found to be much more const. than that of either of the other combinations. The variation was found to be due to the diffusion of the material in the salt bridge into the cell. Taking the normal calomel cell at 25° as 0.5648 volt, the value of the satd. electrode is 0.5266 v. and the temp. coeff. is +0.0002 v. per degree rise in temp. between 5 and 60°. One of the chief advantages of the cell lies in the fact that it is much less sensitive to slight changes in the concn. of the KCl than the cells containing more dil. KCl solns. It is easily reproducible and can be relied upon for constancy over long periods of time. Many exptl. data are given.

C. R. PARK

The standardization of weights. A. J. HOPKINS, J. B. ZINN AND HARRIET ROGERS. *J. Am. Chem. Soc.* 42, 2528-31(1920).—Instead of using one standard weight as a basis for comparison in standardizing a set of weights, a modification is being followed by students in Amherst College, consisting of the introduction of checks at fixed intervals in the progress of standardization. The advantages are (1) the utilization by students of single hrs.; (2) greater ease of understanding, and (3) the redistribution of errors over shorter ranges, thereby avoiding multiplication of errors.

J. M. B.

Commercial photometry. A. L. POWELL AND J. A. SUMMERS. *Gen. Elec. Rev.* 24, 50-8(1921).—A review. A very useful bibliography is appended.

C. G. F.

Methods for the production and measurement of high vacua. S. DUSHMAN. *Gen. Elec. Rev.* 24, 58-68(1921); cf. *C. A.* 15, 193.—Discussion of the absorption of gases by charcoal and Pd black.

C. G. F.

Tellurium and bromine at the time of their discovery. PAUL DIERGART. *Z. angew. Chem.* 33, 299-300(1920).

E. J. C.

A historical contribution relative to the combustion process. E. O. VON LIPP-MANN. *Z. angew. Chem.* 33, I, 301(1920).

E. J. C.

Photographic copies as substitutes for original literature. ALFRED STROCK. *Z. angew. Chem.* 33, I, 301-3(1920).

E. J. C.

Partial and total pressures of aqueous solutions of $NH_3 \cdot CO_2$. (TERRES, WEISER) 21. Theory of the velocity of chemical reaction (LINDEMANN) 3.

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3—SUBATOMIC PHENOMENA AND RADIOCHEMISTRY

GERALD L. WENDT

Is the atom the ultimate magnetic particle? ARTHUR H. COMPTON AND OSWALD ROGNLEY. *Phys. Rev.* 16, 464-76(1920); cf. *C. A.* 12, 249. A. L. F.

The separation of the element chlorine into normal chlorine and meta-chlorine and the positive electron. F. W. ASTON. *Nature* 106, 375(1920).—A. withdraws his previous statement, admitting that he failed to note that Harkins (*C. A.* 14, 2123) claims a wt. of 1.000 for the H nucleus only when the latter is combined in a complex atom. W. H. ROSS

Atomic structure. NORMAN R. CAMPBELL. *Nature* 106, 408-9(1920).—According to the usual representation of the Bohr-Sommerfeld theory of the structure of the atom the electrons are revolving in planetary orbits, whereas the Lewis-Langmuir or Born-Landé theory indicates that the electrons are fixed in const. positions. It is argued, however, that the conclusions to be drawn from the two theories are not really inconsistent. The Bohr-Sommerfeld theory does not make explicit use of the assumption that the electrons in their stable states are moving, for it is possible that a fixed electron may have the properties which classical dynamics attributes to a moving electron. W. H. ROSS

The constitution of the elements. F. W. ASTON. Cavendish Lab. *Nature* 106, 468(1920).—In continuing the work described in previous papers (*C. A.* 14, 493, 891, 1629, 3014) it has been demonstrated that *iodine* is a simple element of mass 127. Two more probable isotopes of *xenon* have been identified and trustworthy values have been obtained for the at. wts. of the five already found. Additional evidence on *argon* leads to the conclusion that its isotope 36 need no longer be regarded as doubtful. W. H. ROSS

Name for the positive nucleus. OLIVER LODGE. *Nature* 106, 467(1920).—As an aid to the selection of a suitable name for the unit of positive electricity there is suggested, in addition to the name "proton" as proposed by Rutherford, such terms as amhron, merron, uron, prime, centron and hylon. L. expresses a preference for the name last mentioned. FREDERICK SODDY. *Ibid* 106, 502-3(1920).—The question is raised as to the necessity of providing a new name for the H nucleus or unit of positive electricity in view of the meaning of the well known term "hydron." W. H. ROSS

The dimensions of atoms. A. O. RANKINE. *Phil. Mag.* 40, 516-9(1920).—The diams. of atoms of the inert gases and of the halogen gases and of N and O as calcd. from viscosity measurements are compared with the values recently assigned by W. L. Bragg as result of calcs. based on X-ray crystal measurements. In all cases the diam. of the free mol. of the gas appears somewhat larger than in the crystal. This probably means that the approach of the outer electrons of two mols. is not so close in the gas as in the "close packed" crystal structure. S. C. LIND

Cubic atoms, the periodic system, and the structure of molecules. A. LANDÉ. *Z. Physik* 2, 380-404(1920); cf. *C. A.* 14, 1481, 2284, 3571.—Of the possible quantized arrangements of electrons, that in which there are two electrons 75° apart rotating in each of the four planes perpendicular to a diagonal of a cube, is shown to be more stable than any other arrangement of 8 electrons, and that neglecting mutual disturbances, the most stable configuration for an atom of more electrons is a series of cubic "shells," at least for large values of the nuclear charge. The approximate value of

the energy of a cube of 8 electrons is $(8Nh/n^2)(Z - 2.486)$, where N is the Rydberg constant, n an integer and Z the nuclear charge. Ionized alkalis would have the cubic arrangement, but for the halogens others are as stable. For oxygen two shells of four electrons each are dynamically more stable than a single cube of 8. A H^+ nucleus can be in equil. at a corner of a cube, which makes possible the calcn. of the heat of ionization of HF and H_2O , which are found to be 350 and 533 kg. cal./mol., resp. Values for the radii are in agreement with values estd. in other ways. There is a summary of preceding papers.

F. C. HOYT

The structure of the hydrogen molecule and the hydrogen ion. IRVING LANGMUIR. *Science* 52, 433-7(1920).—It is conceived by L. that the H_2 mol. has a structure similar to that of He already described in a former paper (cf. *C. A.* 14, 2580) except that there are 2 nuclei. The electrons move in sep. orbits in a plane which is perpendicular to and bisects the line connecting the nuclei. The electrons are at any time situated symmetrically with respect to another plane which passes through both nuclei. Each electron oscillates along a nearly semi-circular path. By means of classical mechanics it is calcd. that the total energy W of the mol. is equal to $(1.604 W_0 a_0)/a$ where W_0 is the corresponding energy of the H atom according to Bohr's theory, a the distance from the center of the mol. to the mid-point of the orbit, and a_0 the radius of the electron orbit. In the H atom, $a_0 = 0.530 \times 10^{-8}$ cm. It should be possible by means of the quantum theory to det. a and fix the abs. dimensions of this model, but so far no general method of detg. what system of coördinates should be used suggests itself. The value of a is calcd. from the known heat of dissociation of mol. H_2 into atoms, and this result is tested by calcg. other properties of H_2 . Taking q the heat of dissociation at const. vol., as 84,000 cal. per g.-mol., it follows that $W/W_0 = 2.270$, and hence $a = 0.375 \times 10^{-8}$ cm. When the electrons are at the ends of their orbits their distance from the center is 0.432×10^{-8} cm. In Bohr's model for the H_2 mol. the radius of the orbit of the electrons is 0.506×10^{-8} cm. In the new model the distance of the nuclei from the center is 0.232×10^{-8} cm. and the moment of inertia of the mol. about its center is 1.78×10^{-41} g. cm², while for the Bohr model these values are, resp., 0.292×10^{-41} and 2.81×10^{-41} . From the theory of band spectra recently developed by Lenz, Heurlinger, and others, Sommerfeld has calcd. that the moment of inertia is 1.85×10^{-41} g. cm². It is also shown that it is possible to construct a model of the positive H_2 ion which is stable, by assuming that the electron oscillates along a rectilinear path passing through the center of the ion and perpendicular to the line joining the nuclei. Applying the quantum theory by two different methods, the energy of the ion is calcd. to be $1.522 W_0$ and $1.229 W_0$, and the corresponding ionizing potentials 10.15 and 14.1 v., resp. The first is in fair agreement with the exptl. values (11 to 11.5 v.).

A. L. FENN

Radium content of the rocks of the Loetschberg tunnel. J. H. T. POOLE. *Phil. Mag.* 40, 466-71(1920).—The Ra content was detd. by the electroscopic emanation method after pulverizing the rocks and decomp. them with $Na_2(K)CO_3$ mixt. In calcareous rocks borax and boric acid were added also to assist the decompn. The total length of the tunnel is 14.5 km., starting at the northern terminal near Kandersteg 4 km. passing through Jurassic limestone, 6.5 km. through Gastern granite and then 4.2 km. of cryst. schist of all classes to the southern terminus. The specimens were not uniformly distributed, most being taken from the schist. No marked variation in Ra content was found along the length of the tunnel, the highest being 6.5×10^{-13} g. per g. in quartz schist and the lowest about 0.8×10^{-13} g. per g. in talc. schist. The Ra content according to rocks shows anhydrite (7 specimens) to be lowest with 1.1×10^{-13} , limestone next lowest with 1.5, while hornblende schist and schist luster are highest with 3.1 and 3.4×10^{-13} g. per g., resp. The Gastern granite averages $2.3 \times$

10^{-18} or neglecting one low value 2.5, in good agreement with values for granites previously examd.

S. C. LIND

Determination of charges on fog particles; the existence of a subelectron. ERNST RADEL. *Z. Physik* 3, 63-88(1920).—R. repeats with some changes the expts. of Millikan and Ehrenhaft (*C. A.* 13, 1416) on the motion of a charged particle in an elec. field as a means of detg. its charge. Using Stokes' law for the charge and radius the smallest charge obtainable agrees well with Millikan's value for the charge of the electron in the case of particles of oil, rosin, and Hg of radius from 10^{-4} to 8×10^{-4} cm. For Hg particles of smaller radius and for Au particles the charge is a multiple of a smaller "quantum." Using the Einstein equation for the charge, which is independent of the radius, however, results are obtained which show no systematic deviation from Millikan's value. R. concludes that there is no real evidence for the existence of a "subelectron."

F. C. HOTT

The nature of the carrier of the anode rays. G. P. THOMSON. *Proc. Cambridge Philosophical Soc.* 20, 1, 210-1(1920).—A short report of preliminary expts. in which a paste made by mixing KI, LiCl and graphite is used as the anode of a discharge tube. The app. is designed to produce a narrow beam of rays, which is deflected by electrostatic and magnetic fields and then allowed to strike a photographic plate, as in positiveray analysis. The parabolas formed on the photographic plate indicate the presence of Li and K in the anode stream, but the method is not yet refined enough to detect small proportions of isotopes which may be present.

ARTHUR COHEN

The collisions of electrons with molecules of a gas. J. S. TOWNSEND. *Phil. Mag.* 40, 505-11(1920).—Referring to a recent paper by Pedersen (*C. A.* 14, 3581) on this subject, in which the theory was put forward that each collision reduces the velocity of the electron to zero, T. points out several errors in Pedersen's work and goes on to show what proportion of energy is lost by an electron on colliding with a mol. For example, in air at a pressure of 1 mm. and with a force of 20 v. per cm. applied, the electron makes 240 collisions per cm., the mean final velocity corresponding to a potential of 2.1 v. So that in the final steady state of motion the energy corresponding to a potential fall of 20 v. is dissipated in 290 collisions so that the av. loss is $1/14.5$ v. per collision or about 3% of the whole energy or 1.5% of the velocity at a velocity of 8.5×10^7 cm. sec.⁻¹. The elasticity of the impact varies with the velocity; for low velocity the elasticity may be considered as perfect.

S. C. LIND

Arcing voltages in mercury vapor as a function of the temperature of the cathode. T. C. HEBB. *Phys. Rev.* 16, 375-86(1920); cf. *C. A.* 14, 2582.—Using an improved app. designed to produce a uniform Hg vapor atm., a linear relation was observed between the striking voltage and the current through the cathode for the larger currents. For a Pt cathode covered with CaO this is shown to mean that the striking voltage bears a linear relation to the cathode temp. in the range 1630° to 1925° K. The temp. was calcd. from the observed elec. resistance of the Pt cathode strip, assuming a linear temp.-resistance relation above 1100°. The results suggest that the difference between the potential at which ionization takes place and the accepted ionization potential is directly proportional to the abs. temp. The lowest striking voltage obtained with a CaO-coated Pt cathode was approx. 4.9 v. With a W cathode, uncoated, a striking voltage as low as 3.2 v. was obtained. A thinly coated Pt cathode produced an arc at 6.0 v. whereas a thickly coated one produced an arc at 4.9 v. A hot anode used with a thinly coated Pt cathode produced an arc at a lower voltage than when the anode was not heated. The results are briefly discussed but no definite theory is offered to explain them.

A. L. FIELD

Variation with pressure of the residual ionization due to the penetrating radiation. K. MELVINA DOWNEY. *Phys. Rev.* 16, 420-37(1920).—The app. was compensated for fluctuations of the battery furnishing the high potential required for satn. as follows:

The ionization chambers were 2 cast-steel spherical shells, 1 ft. inside diam. and 1 in. thick. The central rod electrode of each sphere was connected to the fiber of a string electroscope of the Lutz-Edelmann type. The spheres were connected to the opposite ends of a 10 megohm resistance. The mid-point of this resistance was connected to the earth terminal as were also the guard rings, shielding tube, and electroscope case. Connections were made at the end terminals of the 10 megohm resistance to the high-potential source (700 v. for the residual ionization and 1500 v. for the ionization due to rays from RaBr). The 2 parallel plates of the electroscope were connected by a megohm, the mid-point of which was earthed and the ends of which were connected to the terminals of a 100-v. battery. The sensitivity of the electrometer was maintained at 10 divisions per 1 v. The ionization chamber was a cylindrical vessel having a vol. of 6000 cc. The 2 hemispherical parts of each steel sphere were bolted together along a flange, the joint being made air-tight by means of a mixt. of resin and beeswax applied hot. The rate of deflection of the electroscope fiber was detd. by the excess ionization resulting from the increase of pressure in the sphere A above the atm. pressure maintained in the sphere B. The variation in the natural ionization with pressure was found to obey a linear law up to 21.5 atm. This indicates that the ionization within a closed vessel is not due to a soft radiation, but is detd. either directly by a penetrating radiation of the γ type or by a hard corpuscular radiation emitted from the walls of the vessel by the penetrating radiation. If attributable to the corpuscular radiation, this radiation itself is then so penetrating that it is not appreciably absorbed in passing through a 1-ft. thickness of air at 20 atm. pressure, equiv. in absorbing power to 20 ft. of air at 1 atm. pressure. The ionization in air due to the γ -rays of Ra was measured for pressures ranging up to 40 atm. From 4 to 20 atm. a linear relation existed. A change in slope at 20 atm. to a value about 0.6 that at lower pressures may be attributed to some of the β -rays having completed their paths. At the high pressures there was a suggestion of an effect due to the secondary radiation from air. A few curves obtained during the preliminary observations indicated the existence of a *diurnal variation* of the penetrating radiation, provided that the sky was not cloudy.

A. L. FELD

Ionization and production of radiation by electron impacts in helium investigated by a new method. K. T. COMPTON. *Phil. Mag.* 40, 553-69(1920); cf. *C. A.* 14, 2582.—A method is described by which it is possible to distinguish between the ionization and radiation produced in He by electronic shock. An electrically heated W cathode is employed as the source of electrons, which are accelerated toward a Pt gauze by a certain potential difference. Those passing through the gauze are retarded by a potential sufficient to prevent their reaching the electrode. The electrode is connected with a quadrant electrometer and may gain positive charge either from positive ions resulting from ionization of the He, or as a result of the photoelec. emission of electrons from the electrode by ultraviolet radiations set up by electronic impacts. The two causes of charge are distinguished by using a cylindrical electrode which can be turned so as to present either an end closed with a Pt foil, or one with a Pt gauze. For the reception of positive ions, it is indifferent which end is presented, but in the case of ultraviolet radiation, a large part passes through the gauze and into the gas and does not result in the loss of electrons from the cylinder as is the case when the foil is presented. The result shows that resonance radiation sets in at 20.2 v. and ionization at 25.5 v. Ionization is also observed between 20.2 and 25.5 v. in proportion increasing with the gas pressure and the bombarding current density, which ionization is believed to be a secondary effect due to impacts with electrons containing absorbed radiant energy of the resonance radiation from neighboring atoms. This method of ionization appears to be very important at high gas pressures.

S. C. LIND

The relative ionization potentials of gases as observed in thermionic valves. G. STEAD AND B. S. GOSSLING. *Phil. Mag.* 40, 413-26(1920).—Some of the earlier methods of detg. ionization potential have been criticized as indicating not the appearance of positive ions, but some other effect, such as photoelec. emission of electrons from the electrodes intended to collect the positive ion when formed. It is believed that this source of error is eliminated by use of the thermionic valve. The tube used was a three-electrode system, the cold electrodes being connected together. Next the W filament was a spiral Mo wire of 0.4 mm. diam. coiled into a helix of diam. 4.5 mm. having 4 or 5 convolutions per cm. Outside this was a Ni cylinder 10 mm. in diam. The uncorrected results are the following: Hg vapor 10.8 v., A 12.5, H 15, CO 15, N 17.2, He 20.8. The potential differences given are between the anode and the negative end of the filament and, therefore, represent maxima. A correction of about 0.3 v. is indicated as the one which should be subtracted from the uncorrected ionization potentials. A further correction for the initial velocity of the electrons has the opposite sign and a value of about 0.4 to 0.5 volt which a little more than compensates the first correction. Comparison of these results with those for the ionization potential as detd. by various authorities is made.

S. C. LIND

The effect of a trace of impurity on the measurement of the ionization velocity for electrons in helium. FRANK HORTON. *Phil. Mag.* 40, 440-50(1920).—H. has used various forms of thermionic valves to det. the voltages at which radiation and ionization could be produced in pure He, and to study the effect of impurities, especially of Hg vapor on the limiting values. The results indicate that ionization in pure He does not occur below 25.0 v., a value slightly lower than that obtained by Horton and Davies (C. A. 13, 1669). The presence of small amts. of Hg vapor enables ionization to be detected as low as 20 v.; above 21 v. the radiation from He predominates, and at 23.7 v. the ionization of Hg constitutes only a small part, and the enhancement of the Hg lines at this voltage must be due to the largely increased amount of He radiation.

S. C. LIND

The disappearance of gas in the electric discharge. N. R. CAMPBELL AND J. W. H. RYDER. General Electric Co. of London. *Phil. Mag.* 40, 585-611(1920).—An attempt is made to explain the disappearance of gas in elec. discharge at low gas pressure. Particular attention is paid to the elec. "clean up" as distinguished from the chem. one already studied in great detail by Langmuir. The disappearance of gas appears closely related to the appearance of the elec. glow discharge. The glow is accompanied by a sudden increase of current. The electrical conditions of the glow are described. It appears sharply at a given potential, dependent upon the pressure and the cathode temp., called the glow potential. The glow potential is considered with reference to thermionic emission, gas pressure, nature of the gas, and form of the electrodes. Small quantities of impurities greatly change the glow potential, especially in H. The theory of the glow is deferred for later discussion, but the general connection between the appearance of the glow and ionization is considered, as well as the importance of the glow potential for interpreting the rate of disappearance of gas. Expts. on the disappearance of CO in the glow discharge are discussed in detail. An explanation proposed is the assumption of reversible chemical action. One of the interesting actions indicated is the elimination of C from 2 CO to form CO₂, which is condensed in the liquid-air trap. There is also evidence of the reversibility of this action. The disappearance of other gases will be discussed in a sequel.

S. C. LIND

The pressure on the poles of an electric arc. S. RAYNER. *Phil. Mag.* 40, 511-2(1920).—Note with reference to an article of the same title by Duffield, Burnham and Davis (C. A. 14, 3358). R. shows that while ions moving toward an electrode of opposite sign cannot produce a pressure on it since the pull on the electrode is balanced

by the impact of the ion on reaching the electrode, ions of the same kind moving from an electrode produce an effect not balanced by any other force, which will result in a strong pressure in the direction of the electric field, and in setting up an "electrical wind" by motion of the surrounding air. By use of a vane it was shown that a current of 10^{-6} amp. through the gas produces a pressure of at least 25 dynes per cm^2 . In an elec. arc where several amps. pass through the gas a pressure would be expected that would crush the C poles. Apparently the electric wind pressure does not continue to rise with the current, but actually falls, an observation previously made by R.

S. C. LIND

The ultra-micrometer: an application of the thermionic valve to the measurement of very small distances. R. WHIDINGTON. *Phil. Mag.* 40, 634-9(1920).—Interferometer methods for the measurement of small distances are limited to the wave lengths of the light producing the fringes. The present method is applicable to distances of the order of magnitude of the at. diam. A circuit consisting of a parallel plate condenser and inductance is maintained in vibration by a thermionic valve. Small changes in the distance sepg. the plates produce a change in the frequency of the oscillations which can be accurately detd. by methods described in the original. Changes as small as $1/100$ millionth of an inch can be easily detected. Further refinement will probably lower this limit considerably.

S. C. LIND

Soft X-rays. H. M. DADOURIAN. *Phys. Rev.* 16, 481-5(1920).—Reply to criticism by Laird (*C. A.* 14, 2583) of a former paper by D. (*C. A.* 14, 2128).

A. L. F.

Continuous X-ray spectrum. HERMANN BEHNKEN. *Z. Physik* 3, 48-59(1920).—B. confirms the quantum law as applied to the general radiation, using for the voltage the max. voltage of an alternating current. Changing the current through the tube in the ratio of 1:6 is without effect on the critical voltage, position of the focal spot, or energy distribution.

F. C. HOVR

Doublets of the K-series of X-rays. NILS STENSSON. *Lund. Z. Physik* 3, 60-2(1920).—S. measures with a vacuum spectrograph the doublet difference $\alpha_1 \alpha_2$ from Cl to Cu. The agreement with Sommerfeld's value calcd. from the relativity correction is good to within about 10%, the observed values being consistently higher. The $K_{\alpha_1 \alpha_2}$ doublet, unsepd., is measured over the same range. A spectrograph with high dispersion is to be built.

F. C. HOVR

Scattering of X-rays. KARL HERMANN. *Physik. Z.* 21, 534-41(1920).—H. modifies the work of Debye (*Sci. Abs.* 18, 498) to make it apply to the case where the electron rings are all parallel, as under the influence of a magnetic field. In this way he shows that a magnetic field ought to produce small effects on the Laue diffraction spots. Mechanical pressure may also produce an orientation.

F. C. HOVR

Optical rotation, optical isomerism, and the ring electron. H. STANLEY ALLEN. *Phil. Mag.* 40, 426-40(1920).—A. adopts the Parsons magneton theory of the electron as a basis of a theory of optical rotation. Such a ring electron vibrating in a linear path takes the place of an ordinary electron moving in a spiral path as postulated by Drude. A. shows that rotation of the plane of polarization of light will result, and finds an expression for the amt. of rotation per unit length. The octet theory of at. structure due to Lewis and Langmuir is employed by A. to obtain a graphical representation of dextro- and levo-rotatory compds. The exptl. facts are in good agreement with the theory, which may be applied not merely to C compds. but to any compd. containing an asymmetric atom.

S. C. LIND

Electric doublets in liquid dielectrics. J. HERWEG. *Z. Physik* 3, 36-47(1920).—The assumption of Debye (*Sci. Abs.* 15, 184) of the presence of free electric doublets leads to a decrease of the dielec. const. with a strong electric field. H. measures a decrease of 10^{-4} in a field of 280 v./cm., agreeing with his calcd. value.

F. C. HOVR

Photoelectric conductivity and phosphorescence. B. GUDDEN AND R. POHL. *Z. Physik* 3, 98-103(1920); cf. *C. A.* 14, 3363.—The elec. cond. of phosphorescent Ca, Bi, and Na under field strength of 16,000 v./cm. as a function of the wave length of the exciting light is measured. The two maxima in the neighborhood of 450 $\mu\mu$ correspond exactly to the two maxima observed by Lenard in the phosphorescent spectra of these substances. The cond. of a substance showing persistent phosphorescence is increased by illumination. F. C. HOYT

Absorption spectra of hydrogen chloride. F. W. LOOMIS. *Nature* 106, 179-80(1920).—A short note suggesting that the satellites observed by Imes (*C. A.* 14, 3366) beside each line in the HCl absorption band at 1.76 μ may be due to the heavier of the isotopes of Cl. A detailed account of further measurements on these lines together with a theory of their formation is to be published by Loomis. ARTHUR COHEN

Theory of the rotation spectrum. FRITS REICHE. *Z. Physik* 1, 283-93(1920).—The rotation of the entire mol. of a diatomic gas with frequency ν_r has the effect, as shown by Rayleigh, of combining with a frequency ν_0 of vibration of the atoms within the mol. to give frequencies $\nu_0 \pm \nu_r$. Planck (*C. A.* 12, 1147) has used $\nu_r = nh/4\pi^2 J$ where J is a moment of inertia, giving a series $\nu = \nu_0 \pm nh/4\pi^2 J$. R. tries to generalize this by treating the mol. as a symmetrical "top" with two moments of inertia and gets the energy of the whole mol. to depend on four quantum integers. Limiting the number of possible transitions, by the selection principle this leads to a series of the form $\nu = \nu_0 \pm (n+1)h/4\pi^2 J$, which is in agreement with Imes' measurements (*C. A.* 14, 3366) as is also Planck's result. F. C. HOYT

The helium-hydrogen series constants. HERBERT BELL. *Phil. Mag.* 40, 489-94(1920).—A discussion of measurements by various authorities of the spectral lines of H and He and of the formulas expressing the values as corrected on different assumptions. The need of interferometer measurements on the earlier H lines is pointed out. S. C. LIND

Deduction of Rydberg's law from the quantum theory of spectral emission. SATYENDRA NATH BASU. *Phil. Mag.* 40, 619-27(1920).—A mathematical deduction of the laws governing the spectral series of the elements, based on Bohr's quantum theory of spectral emission. S. C. LIND

Scattering of light by unsymmetrical atoms and molecules. J. J. THOMSON. *Phil. Mag.* 40, 393-413(1920).—When the arrangement of electrons in an atom is symmetrical, the displacement of an electron by an external force will always be in the direction of the force, and the ratio of the displacement to the force will be independent of the direction of the force. The same conditions do not hold when the atom is unsymmetrical and the displacement of an electron is not in the direction of the force. The laws of scattering are then treated mathematically. The law of the inverse square of the distance is discussed with reference to its applicability to α -particles. The idea is advanced that it applies at the very short distances represented in α -particle scattering and again at greater distances, while in between at distances at which c (distance of electron from center) is of the same order as r (radius of the atom) an entirely different law may apply. S. C. LIND

Ionization in the solar chromosphere. MEGH NAD SAHA. *Phil. Mag.* 40, 72-88(1920).—From a discussion of the high-level chromospheric spectrum of the sun it is shown that the region is composed chiefly of ionized atoms of Ca, Ba, Sr, Sc, Ti and Fe. The enhanced lines are the same ones which are brightest in the spark spectrum, which can best be explained by ionization. In the lower layers both ionized and neutral atoms occur. An attempt is made to account for this on the basis of Nernst's theory of the "reaction isobars" by assuming that ionization is a reversible process according to the equation $\text{Ca} \rightleftharpoons \text{Ca}^+ + e \rightleftharpoons U$, U , the energy of ionization, can be calculated from

the ionization potential of the elements. For the detn. of Nernst's *chem. const.* and *sp. heat* the electron is assumed to be a monatomic gas of at. wt. 1/1836. A great influence of pressure on the relative degree of ionization is marked. Almost complete ionization of Ca, Sr and Ba atoms in the high-level chromosphere is due to the low pressure. H_2 is shown to be completely dissociated into atoms at all points in the solar atm. It is also shown that the higher the ionization potential of an element the more difficult will be its ionization thermally. Calcs. show that H and He cannot be appreciably ionized anywhere in the sun. Hc can be ionized only in the stars having the highest temps. ($> 16,000^\circ K$). S. C. LIND

The relation of radiant energy to matter, space and time. J. D. ROSS. Seattle. *J. Am. Inst. Elec. Eng.* 39, 985-94(1920).—It has been shown experimentally that: (1) A reflected beam of light is not a continuation of the incident beam, there being an intermediate transformation of energy, light to elec. current, and *vice versa*. (2) A moving magnetic line of force and a light ray are identical and their direction of propagation is the same, the latter not traveling in straight lines (except when of very short wave length) but in closed circuits tending to come back to the starting point. (3) Very long light waves obey Oersted's law instead of the law of inverse squares. (4) The phenomenon of the repulsion of light is identical both with the repulsion of unlike magnetic poles and with the repulsion between the primary and secondary currents of a transformer. (5) The kinetic theory of heat is untenable. The Brownian movements of suspended particles prove intense vibration in the particles of matter, but this vibration is the effect, not the cause of heat. (6) An elec. current is not the direct source of a magnetic line. The electrostatic and electromagnetic fields of a changing elec. current are produced progressively, not simultaneously, the former being produced first and the latter being in turn produced from the former. Conversely, a magnetic line does not generate an elec. current, except through the medium of the electrostatic field at right angles to it. (7) Very long light waves may be generated at a point and conducted along magnetic substances as a core or guide. (8) Except in cases of selective absorption the efficiency of a reflector is the same as the elec. cond. of the material from which it is made. From the foregoing and the results and theories of others, assumptions are deduced relative to: the velocity of light; the opposition to light in space; the nature of X-rays, the ether, gravity, and of matter at abs. zero; the structure of space; the formation of matter. H. JERMAIN CREIGHTON

Note on the theory of the velocity of chemical reaction. F. A. LINDEMANN. *Phil. Mag.* 40, 671-4(1920).—A criticism of Lewis' *radiation theory of chem. action*. For the inversion of sucrose, which according to Lewis should be produced by a radiation of $\lambda = 1.05 \mu$; L. calcs. that radiation density of this wave length from the sun would be at least 10^{13} greater than that in the dark, and the absence of a photochem. effect of sunlight on the reaction appears to invalidate Lewis' theory. S. C. LIND

Ignition of gases at reduced pressures by transient arcs (THORNTON) 24. Removing gases from vacuum apparatus (Brit. pat. 151,611) 1.

BEIN, W.: Das chemische Element, seine Wandlung und sein Bau als Ergebnis der wissensch. Forschung. Berlin: Walter de Gruyter & Co. M. 45, bound M. 53.

BORN, MAX: Der Aufbau der Materie. Drei Aufsätze über moderne Atomistik und Elektronentheorie. Berlin: Julius Springer. 81 pp. M. 8.60. For review see *Nature* 106, 339(1920).

MAYER, S. AND SCHWEIDLER, E. R. v.: Radioaktivität. Leipzig: B. C. Teubner. 540 pp. 170 M. For review see *J. Am. Chem. Soc.* 43, 213(1921).

SILBERSTEIN, L.: Report on the Quantum Theory of Spectra. London: Adam Hilger. 42 pp. For review see *Phys. Rev.* 17, 71(1921).

SUTER, P.: Die Elektronentheorie der Metalle. Bern: Haupt. M. 12.

4—ELECTROCHEMISTRY

COLIN G. FINK

Electrochemistry in 1920. A. A. GRAY *Elec. Rev.* 78, 9-11(1921).—A short review. C. G. F.

Booth rotating electric furnace. CARL H. BOOTH. *Iron Age* 106, 1463-4(1920); *Foundry* 48, 994-7, 1004.—See *C. A.* 14, 495; 15, 23. C. G. F.

Electric furnace with repelling arc. F. VON SCHLEGEL. *Iron Age* 106, 1556-7(1920).—A more detailed account than that referred to in *C. A.* 14, 3596. C. G. F.

Heat losses through electrodes in electric furnaces. FRANK HODSON. *Iron Age* 106, 1262-3(1920).—See *C. A.* 15, 22. C. G. F.

Electric furnace usage fast increasing. ANON. *Elec. World* 77, 77-9(1921).—A short review of the electrochem. and electrothermal industries during 1920. On Jan. 1, 1921, there were in U. S. 765 steel and non-ferrous elec. furnaces installed of a total connected load of 415,000 kv. a. The av. size steel furnace in U. S. is 3 tons. C. G. F.

The present status of the electric furnace in the American metal industries. ROBERT M. KEENEY. *Chem. Met. Eng.* 23, 980-4(1920); cf. *C. A.* 14, 1935, 1936. —Use of the elec. furnace developed very rapidly during the war. For pig Fe, elec. smelting of ore is not economical in U. S., but elec. melting up of scrap steel, etc., gives cheap pigs. Transverse strength and S content are improved by refining cupola Fe in basic elec. furnaces, especially for malleable castings. Nearly 400 elec. steel furnaces are in operation using over 600,000 kv. a., of which about 50% is for alloy steel. There is an increasing tendency toward 3-phase Heroult-type furnaces having high power input, low reactance circuits, and automatic electrode control. Heat treating is now largely carried on in C or Ni-Cr ribbon resistor furnaces, which have high power factor and complete temp. control. An enormous expansion of the ferro-alloy industry resulted from elec. furnace installations of over 200,000 kv. a. U. S. led the world in Fe-Si with 100,000 tons per yr.; Fe-W, Fe-Cr, even Fe-V, were largely exported; Fe-Mn is made by elec. smelting, and Fe-Mo widely used in steel. Three-phase furnaces are suitable for Fe-Si, Fe-Mn, and Fe-Cr, on account of lower m. p.; but Fe-W, Fe-Mo, Fe-V, Fe-U and Fe-Ti require single-phase. The voltage should be const. and adequate for proper power input. For melting brass and non-Fe alloys, the use of elec. furnaces has grown rapidly to 23,000 kv. a., of which $\frac{1}{2}$ is in C resistor, $\frac{1}{2}$ arc, and remainder induction types, practically all being single-phase. Elec. smelting on Zn ores is not yet commercial here, although successful in Sweden with cheaper power; a 1000 kw. Zn-melting furnace is reported in operation over a yr. using 70-80 kw. per ton, with only 0.024% metal losses in dross. In many industries elec. furnaces are limited to hydro-elec. power, but increased use may be expected in (1) Fe castings from scrap or a duplex process of cupola melting with elec. furnace refining, (2) steel castings and alloy steels, (3) ferro-alloys, (4) Al, (5) brass melting, (6) electrolytic Zn and (7) elec. smelting of non-ferrous ores. F. H. HOTCHKISS

Power consumption in production of castings in the electric furnace. M. A. COUTAGNE. *Rev. métal.* 17, 450-68(1920).—Exptl. and theoretical data are given on the heats of reduction and fusion of Fe, Fe alloys, Si, Mn, etc. Heat balance sheets of elec. furnaces for smelting Fe ores are tabulated and also compared with the heat balance of the ordinary blast furnace. O. A. HODGSON

Electric versus combustion furnaces for low temperatures. FRANK W. BROOKE AND GEO. P. MILLS. *Chem. Met. Eng.* 23, 1008-10(1920).—The more recent com. applications of elec. energy to low temp. work (up to 1000°) are discussed. The advantages of elec. heat over that from combustion are summarized as: (1) Absolute control of temp; (2) saving in labor (no handling of coal, ashes, oil, etc.); (3) upkeep cost inconsiderable; (4) smaller space required; (5) reduction of rejections in finished product; (6) improved product; (7) ideal shop conditions. The items of temp. control, first cost, maintenance, and cost of operation are considered separately and it is shown that, all things considered, the elec. furnace is frequently the most economical installation.

W. E. RUDER

Economies of melting brass electrically. ANON. *Elec. World* 76, 1253-5(1920).—The operating records of many brass and alloy foundries show that great economy has resulted from the use of small rotating elec. furnaces. A large variety of mixts. can be handled in a day's run. With only an 8- or 10-hr. day, energy consumption can be kept low by charging after last "heat" of day, thus storing up enough heat during the night to avoid preheating. Even with power at 2-3 c. per kw. hr. the great savings in metal losses, crucible costs and in labor show a large margin in favor of the elec. furnace. Cf. *C. A.* 14, 2133-4.

F. H. HOTCHKISS

Yields in the manufacture of cyanamide. CH. PLUVINAGE. *Chimie & industrie* 3, 438-9(1920).—The cyanamide industry has changed little since its start in 1908. The N content seldom exceeds 22.5%, although the theoretical yield from 80% CaC_2 is 26% N. While the impurities (chiefly CaO) in CaC_2 greatly diminish the yield, nitrification is never quite complete. Yields are increased by using pulverized CaC_2 , its even distribution around the electrodes, and the use of refractory linings to prevent heat losses. Care must be taken not to decompose CaCN_2 by excess of heat, i. e., too rapid reaction.

F. H. HOTCHKISS

The fundamentals of the electrolytic diaphragm cell. (Decomposition voltage.) HUGH K. MOORE. *Chem. Met. Eng.* 23, 1011-15, 1072-4, 1125-8(1920).—A detailed study, with numerous curves and tables, of the brine compn. and products during electrolysis in diaphragm cells. The current decompn. and energy efficiency are considered first. Rate-of-flow charts are valuable for comparative purposes under like conditions, but care must be taken not to use them for comparison with cells run under entirely different conditions. The use of cc. per amp. hr. instead of lb. per amp. hr. is recommended for charts of operation. Washing the diaphragm not only saves the cost and labor of installing new ones but a washed diaphragm is actually better than a new one. The importance of proper care of cells is emphasized by cost figures varying from \$10.87 to \$20.67 per ton of salt decomposed. Temp. is also an important factor. The rate of flow varies from 16 l. per hr. at 40° to 20 l. per hr. at 70°. An abs. removal of Na_2SO_4 increases the life of the carbons. In an appendix, M. gives a method for calcg. decompn. voltage.

W. E. RUDER

Chemical efficiency and concentration of caustic and salt from Townsend cells. A. H. HOOKER. *Chem. Met. Eng.* 23, 961-8(1920).—A brief description of the installation at the Hooker Electrochem. Co. Modern Townsend cells are operated with 4 v., 4000 amp.; current density, 100 amp. per sq. ft. of cathode. Use of oil and circulation permit high efficiency, 96 to 97%, with only 125 g. NaOH per l. Characteristic properties of cathode liquors are tabulated. Nine excellent charts have been compiled showing the characteristic properties of cathode liquors, such as: sp. gr. and grams per l. of satd. NaCl — NaOH at various temps.; soly. of Cl in brine; ratio of NaOH to water and NaCl ; concn. of NaCl in NaOH soln. and % of NaCl and NaOH during concn.

O. A. HOUGEN

The measurement of electrolytic resistance using alternating currents. H. F. HAWORTH. *Electrician* 85, 433-5(1920); *Trans. Faraday Soc.*, 1920.—When a. c. is

put through an electrolyte the cell acts as a capacity in series with a resistance, both varying with the frequency, temp., concn. and current flowing. Expts. were carried out to compare true and apparent resistance of cells, as well as effects of above variables on resistance and capacity. Measurements were taken by means of a bridge where the cell was in series with a self-inductance, and a similar self-inductance was put in the adjacent arm for proper balance. H. derives many formulas for actual practice, and concludes that the thickness of the gas film between electrodes and electrolyte varies inversely with temp. and current flowing, and directly with the frequency. The impedance curve of the cell, plotted in correct phase relationship to the current for different frequencies, is a straight line cutting the current axis at the point of infinite frequency, which intercept is the true resistance of the electrolyte. The effect of platinizing electrodes is to produce a spongy Pt deposit capable of absorbing the gas film and reducing its thickness. The resistance variations with frequency are much less with platinized electrodes than with non-platinized, but in any case diln. means a decrease in the effective capacity of the cell.

F. H. HOTCHKISS

Some aspects of electrolysis. GELLERT ALLEMAN. *J. Am. Water Works Assoc.* 7, 882-91(1920).—A general résumé of the electrolytic corrosion of Fe in moist soil through the action of elec. currents. The necessity of protection is emphasized.

D. K. FRENCH

The electrical purification of fumes and gases in explosives manufacture during the war. A. DELASALLE. *Chimie & industrie* 4, 291-316(1920).—Simultaneously with American development of the "Cottrell" system, extensive researches were carried on by a French commission for the elec. elimination of various fumes. The most important application was in concn. of H_2SO_4 . Preliminary trials indicated: (1) a potential difference of 4000 v. per cm. as suitable; (2) that the effect is practically independent of frequency; (3) that the discharge of fume particles occurs only at max. of voltage surge; (4) that fringed anodes offer no advantage in humid gases at low temps.; (5) that anodes should have low, and cathodes high, radius of curvature, the latter also being as smooth as possible; and (6) that stoneware chambers are preferable. Various means tried for producing high voltage d. c. included Delon rotating contacts, commutator rectifiers, and Hg-vapor "Kenetrons." A chimney of tubular cells was finally adopted for com. work, and tried out under many conditions. Application was also made to pyrites grate furnaces with great success. Hygienic conditions are improved by this means, and many valuable products recovered.

F. H. HOTCHKISS

(Porcelain) insulation for high voltage transmission systems. ALFRED STILL. *Elec. Rev.* 78, 54-7(1921).—In this instalment the fundamental laws of the dielec. circuit, dielec. strength of insulating materials, effect of condensers connected in series and the pin type insulator, are discussed.

C. G. F.

Modern methods of manufacturing electric lamps. ANON. *Electrician* 85, 686-7(1921).—A brief illustrated account of the process used in the Willesden works of the Pope Elec. Lamp Co. No new data are given.

C. G. F.

Research on the heating of buried cables. S. W. MELSOM, V. COCKBURN, E. FAUSSETT and E. W. MARCHANT. *Electrician* 85, 738-42(1921).—The heating effect of the elec. current on insulated cables tested under various conditions is discussed.

C. G. F.

Operation of lead oxide lightning arresters. ANON. *Elec. Rev.* 78, 75(1921).

C. G. F.

Fused cements and the electric furnace (BIEP) 20. Electric-furnace refractories (GREAVES-WALKER) 19. Compositions and processes for coating glass surfaces (Brit. pat. 150,598) 19. Cleaning wire (Brit. pat. 151,399) 18. Removing gases from vacuum apparatus (Brit. pat. 151,611) 1.

DETTMAR, G.: Die Beseitigung der Kohlennot, unter besonderer Berücksichtigung der Elektrotechnik. Berlin: Jul. Springer. 112 pp. M. 10. For review see *Tonind-Zig.* 44, 1295(1920).

ESCARB, JEAN: Electrometallurgie du fer et de ses alliages. Paris: Dunod. 811 pp. 90 fr.

ESSELBORN: Lehrbuch für Elektrotechnik. Leipzig: Wilhelm Engelmann. 1230 pp. M. 60, bound M. 72 + 50%. War tax and 20% Sort. For review see *Z. tech. Physik.* 1, 232(1920).

YOUNG, A. P.: The Elements of Electrotechnics. London: Isaac Pitman & Sons. 348 pp. 7s. 6d. net. For review see *Nature* 106, 340(1920).

Primary electric battery. C. B. SCHOENMEHL and M. L. MARTUS. U. S. 1,360,589, Nov. 30. Structural features.

Storage battery. J. BALZANO. U. S. 1,362,554, Dec. 14. Structural features.

Storage battery. E. HACKING. U. S. 1,362,294, Dec. 14. Structural features.

Storage battery. D. A. GRIEB. U. S. 1,362,289, Dec. 14. Structural features.

Storage batteries. G. MARCONI. Brit. 151,293, Apr. 5, 1918. A storage battery consists of C and Zn in a soln. of $ZnCl_2$ together with the chloride of an alkali metal or metal of an alk. earth such as $CaCl_2$ or $NaCl$, the soln. being kept under a pressure of about 20 atms. In an example, the electrolyte consists of a soln. of 500–50 g. of $CaCl_2$ and 400 g. of $ZnCl_2$ dissolved in 600 g. of H_2O . A suitable construction is specified.

Storing accumulators. W. E. HOLLAND and L. J. PEARSON. Brit. 150,985, Mar. 8, 1920. To prevent deterioration of accumulators they are stored without electrolyte, but the negative electrodes and wooden separators are moistened with H_2O , the positive electrodes being dry. The cells are sealed in this condition, electrolyte being introduced when they are required for use. The wood separators are provided with ribs which bear against the positive electrodes or against perforated rubber sheets adjacent to the positive electrodes. The cover of the cell has a filling aperture closed by a plug, containing a perforation, which is sealed during the storage of the cell. Cf. C. A. 14, 899.

Electrolyte for storage batteries. E. HACKING. U. S. 1,362,295, Dec. 14. Pumice is mixed with H_2SO_4 to constitute an electrolyte for storage batteries which prevents shedding of active material and holds the plates in position.

Accumulator electrodes. A. POUCHAIN. Brit. 150,811, June 6, 1919. An electrode for an accumulator consists of a perforated and ribbed C or other non-metallic conducting plate coated with Zn by electro deposition from a soln. containing 5 pts. $ZnSO_4$, 1 pt. $HgSO_4$, and 1 pt. $MgSO_4$. A thin layer of Hg with Mg is stated to be first deposited, and then the Zn. The plate may be given an initial electrolytic coating of Cu.

Galvanic batteries. H. R. PALMER. Brit. 150,848, June 23, 1919. The Zn electrode of a dry cell is in the form of a sinuous ribbon the edges of which face the C plate. Both electrodes are embedded in the C and Mn dioxide mixt., the Zn being sepd. therefrom by an absorbent envelope.

Galvanic battery electrodes. S. O. COWPER-COLES. Brit. 150,597, Jan. 8, 1920. A battery electrode, consisting of a thin cylinder of Zn reinforced by Fe, is made by electrodepositing one of these metals on a mandrel coated with stripping material, and then electrolytically coating or lining it with the other metal. It may be made with one end closed. The stripping material may be wax dissolved in turpentine, and coated with graphite. If the Fe is first deposited, it may be annealed before lining with Zn.

Dry battery. F. P. BAUMANN. Japan 34,983, Sept. 26, 1919.

Dry cell electric batteries. O. E. RUHOFF. U. S. 1,361,499, Dec. 7. An electrolyte mixt. for dry cells is formed of NH_4Cl , ZnCl_2 and a gelatinizing material such as starch and H_2O and a portion of the mixt. is poured into a Zn can. A core of depolarizing materia provided with a spacing or centering device is inserted in the material and the contents of the can are then heated sufficiently to effect setting of the contents to solid condition.

Electrolytic cell. I. H. LEVIN. U. S. 1,360,544, Nov. 30. Structural features.

Electrode for cells producing oxygen and hydrogen. I. H. LEVIN. U. S. 1,360,545, Nov. 30. Structural features.

Electrolytic apparatus of the filter-press type. I. H. LEVIN. U. S. 1,360,541, Nov. 30. The app. is especially adapted for production of O and H.

Electrolytic apparatus adapted for production of oxygen and hydrogen. I. H. LEVIN. U. S. 1,360,542, Nov. 30.

Water-feed attachment for electrolytic cells. I. H. LEVIN. U. S. 1,360,543, Nov. 30.

Apparatus for electric precipitation of suspended matter from gases. E. MÖLLER. U. S. 1,362,123, Dec. 14.

Positive plate for battery of Manchester type. K. OTOMORI, G. MISUMI and KOME ELECTRIC MACHINE WORKS. Japan 35,727, Feb. 3, 1920. Pb ribbons, rolled in the form of a spiral and fitted in the frame, are connected.

Copper plates for process printing. S. O. COWPER-COLES. U. S. 1,362,032, Dec. 14. In the electrolytic production of Cu plates for process printing, the electrolyte is caused rapidly to circulate over the surface of the cathode plates, an anode is placed in the electrolyte and the cathode is reciprocated relatively to the anode during the circulation of the electrolyte.

Casting anodes of ferromanganese. C. G. FINK. U. S. 1,361,036, Dec. 7. A casting of ferro-Mn in the form of a thin plate free from checks or cracks is formed for use as an anode in electrolytic process, by melting ferro-Mn, adding 1.5-5% of Al and then casting the scavenged metal into a graphite mold.

Electrolytic production of permanganate. R. E. WILSON and W. G. HORSCH. U. S. 1,360,700, Nov. 30. Permanganate is continuously produced by electrolyzing an alkali metal carbonate soln. as anolyte with caustic alkali soln. as catholyte and an anode containing Mn, while continually adding alkali metal carbonate soln. and circulating the electrolyte.

Electrolytic production of hydroxides from metals. S. FRY. U. S. 1,361,041, Dec. 7. A metal such as scrap Fe or waste tin plate is used as anode in an aq. soln. of NaCl or other salt of Na, K, NH, or Ca of such strength as will cause the metal to be deposited as hydrated oxide, suitable for use as a pigment.

Electroosmotic treatment of colloids. ELEKTRO-OSMOSE AKT.-GES. (GRAF SCHWERIN GES.). Brit. 151,002, Aug. 28, 1920. Aq. solns. of non-conductive or feebly conductive colloids of mineral, vegetable, or animal origin are purified or concd. by an elec. current. There may be added a weak acid or base, with which the H_2O migrates under the action of the current. Mineral acids, monobasic or polybasic aliphatic acids, aromatic acids, NH_3 , alkylamines, alkyl ammonium bases, and their substitution products may for this purpose be added so as to bring the liquid to about decinormal strength. Other electrolytes are previously removed by current from electrodes behind amphoteric or other diaphragms. In the subsequent concn. or purification, the diaphragm through which the H_2O passes should be pervious to H_2O and the alkali or acid residues, while the other is impervious to the colloids. A suitable construction is specified.

Electrolytic etching. J. H. WEEKS. U. S. 1,362,159, Dec. 14. In electrolytic etching, the anode carrying a resistant image to be etched, e. g. a plate of Cu, brass,

steel or Zn, is treated in an electrolyte containing FeSO_4 and other salts or acids which will produce a durable soln. of good conductivity, *e. g.* H_3PO_4 , NaCl , NH_4 oxalate, $\text{K}_2\text{Cr}_2\text{O}_7$ or $(\text{NH}_4)_2\text{SO}_4$ and H_2SO_4 .

Removing liquid from organic and inorganic substances. BOTHO SCHWERIN. Can. 206,814, Dec. 21, 1920. The substance is subdivided as finely as possible in the presence of an electrolyte suitable for bringing the solid body into sol. condition and then subjected to electroosmotic treatment. *Peat* may be thus treated in the presence of NaOH .

Producing endothermic gas reactions. R. PFAEHLER and I. HECHENBLEIKNER. Can. 206,803, Dec. 21, 1920. A current of air to be treated is passed through an elec. arc and a cooling gas is introduced in sep. opposed streams which flow on opposite sides of and into the zone of max. heat of the arc.

Electric furnace for producing endothermic gas reactions. R. PFAEHLER and I. HECHENBLEIKNER. Can. 206,802, Dec. 21, 1920. App. for operating the process of Can. pat. 206,803 (preceding pat.).

Electric furnaces. L. W. WILD and E. P. BARFIELD. Brit. 150,793, June 4, 1919. Molten NaCl for hardening steel is contained in a pot which is made of a metal having a coeff. of expansion approximating to that of the salt and is placed loosely within the SiO_2 wall of a resistance heater. A suitable material for the pot is wrought Fe, well hammered to reduce its porosity. The pot may rest on a block of firebrick within a metal tray, while the resistance heater is suspended from the rim of the pot by means of rings of asbestos and Fe resp. Similar rings close the lower end of the heater, which comprises two SiO_2 cylinders, a resistance winding around the inner cylinder, and non-conducting powder. Cf. 112,731 (C. A. 12, 1151). According to the Provisional Specification, the pot may be made of an alloy of Ni, Cr and Fe.

Electric resistance; adhesives. HUNGARIAN ELEKTHERMAX Co. Brit. 150,673, June 28, 1920. A resistance for an elec. heater is embedded in a yielding insulating fireproof material, which is secured by a thin layer of adhesive to the surface to be heated. The resistance wire is secured to a layer of thin asbestos paper or other felt-like material, from 0.1 to 0.5 mm. thick and covered with a similar layer. The two layers are stuck together by an adhesive consisting of equal parts of H_2O and water glass containing in suspension finely ground clay, kaolin, asbestos powder, etc. The embedded material is then secured by a similar adhesive to the surface to be heated. A thicker layer (about 5–30 mm. thick) of asbestos powder or sand may then be pressed on in a mold and baked. One layer of asbestos paper may be replaced by a layer of adhesive placed directly on the surface to be heated.

Electric insulation. BRITISH THOMSON-HOUSTON Co. Brit. 151,667, Apr. 4, 1919. Insulating material, such as paper or cotton, has part of its moisture of constitution replaced by insulating fluid, such as oil, without loss of mechanical strength and so that the voltage at which it will be punctured increases with increase of temp. Paper is dried by baking by the usual methods, and then immersed in sep. layers in oil at a temp. of from 105 – 10° for 48 hrs.; and to prevent contact with air, the paper should be wound onto reels and thence onto the conductor while still in the oil, the usual lead sheath being drawn over the insulated conductor as it emerges from the oil bath. Cf. 10,543, 1891, 5,165, 1911, (C. A. 6, 2210) and 108,090.

5—PHOTOGRAPHY

L. DERR

The action of phosphorus during oxidation on the photographic plate and its ionizing power. W. P. JORISSEN. Univ. Leyden. *Rec. trav. chim.* 39, 429–34 (1920).—

Bredig and Fensel (*Arch. wissenschaft. Photograph.* 1, 33(1899)) pointed out that P as well as U produces an effect upon the photographic plate that penetrates opaque objects. Barus (*Smithsonian Contrib. to Knowledge* (1901)) made other observations and later J. (*Chem. Weekblad* 1, 237(1904)) without knowledge of earlier work made similar observations. Tzentnershver and Petrikaln (*C. A.* 6, 3355) ascribed the effects observed to the products of the oxidation suspended in the air and suspected that H_2O_2 may be a factor. J. discusses these results and states that these negative results may have been due to too low temp. (the maximum effect occurs at 20–30°, while no effect is observed at 9°) or to the manner of aspirating the app. Thus J. and v. Ressema (*Chem. Weekblad* 6, 935(1909)) observed a diminution or increase in the cond. of the air depending on the velocity and manner in which the fog was eliminated, measuring the increased ionization electrometrically. The expts. of C. and P. were then repeated and the effect on the photographic plate was found to be modified in the same manner as the ionization of the air by changes in the air currents. E. J. WITZEMANN

Production of panchromatic sensitiveness without dyes. J. G. CAPSTAFF AND E. R. BULLOCK. *J. Franklin Inst.* 190, 871–4(1920).—Ordinary plates are rendered panchromatic by use of a $NaHSO_3$ bath followed by a lengthy washing prior to exposure. Apparently the Ag salt is partially reduced to colloidal Ag which may act as an optical sensitizer. The time required for washing was reduced, and the sensitiveness increased by use of a bath of dil. alkali, e. g., 0.1 N Na_2CO_3 or 0.2% $KHCO_3$. The effect is apparently due to the H_2SO_3 content of the $NaHSO_3$, for it is produced by H_2SO_4 and is accentuated by addition of a small amt. of H_2SO_4 to the bisulfite bath, but is not produced by Na_2SO_3 . Presence of an extremely small % of sol. chlorides or bromides in the wash water or the carbonate bath diminishes the effect; thus 0.004% KBr or 0.2% NaCl entirely prevents color sensitizing. The presence of these halide salts in the bisulfite bath is without effect. JOSEPH S. HEPBURN

Graininess in photographic deposits. L. A. JONES AND N. DEISCH. *J. Franklin Inst.* 190, 657–83(1920).—The word "graininess" is used to denote the inhomogeneity of the deposit due to aggregations of particles, and is not merely a matter of size of individual grains. There is a very appreciable difference in the graininess produced by different developing agents, *p*-phenylenediamine, a slow developer, giving very little. Rapidity of development does not necessarily give low graininess, which seems not to be a function of speed of development but rather of some property associated with the particular alkali employed. L. DEER

Developments in practice and art of telephotography. N. TESLA. *Elec. Rev.* 77, 923–5(1920).—The Belin app. consists of two cylinders rotating in synchronism, one for transmitting and the other for reproducing. The former has its surface coated with shellac, a C print of a photograph is wrapped about it, facing the cylinder, and the whole is immersed in hot water. This causes adhesion of the gelatin in proportion to the degree of blackness, thereby producing a likeness in bas-relief. The cylinder carries the stylus of a microphone diaphragm which is slowly moved forward by the revolution of the cylinder. The pressure of the contacts varies with changes of surface and the microphone currents pass over the transmitting wire to the receiving station which registers corresponding deflections of a mirror forming part of a sensitive dead-beat oscillograph. A strong beam of light reflected from the mirror traverses a screen graduated from transparency to opacity and a microscope opening to a sensitive film wrapped around the receiving cylinder. When the operation is completed, development is made as usual. W. H. BOYNTON

KESLER, HEINR.: *Die Photographie*. Berlin: Ver. wissenschaftlicher Verleger, Sammlung Göschen. M. 2.10 + 100% Teuerungszuschlag.

ZIEGLER, WALTER: Die manuellen Techniken Zeichnung, Lithographie, Holzschnitt, Kupferstich und Radierung sowie die verwandten graphischen Verfahren des Hoch-, Flach- und Tiefdrucks. Bd. I. Die Schwarzweisskunst. 3rd Ed. Halle a. S.: Wilhelm Knapp. M. 15, Bound M. 17. For review see *Phot. Rundschau* 57, 215(1920).

Photography. G. W. A. SOSNA and J. E. BRENEBACH. Brit. 11,036, July 29, 1915. Plates and films, the sensitive layers of which have been protected by means of a violet coloring matter such as methyl violet as, *e. g.*, by incorporating the coloring matter in the emulsion, are developed in ordinary artificial light.

Photography. J. AMIOT. Brit. 151,014, Sept. 14, 1920. A sensitive prep. for enabling any surface to be sensitized by a single cold painting or application is obtained by the addition to a soln. of a sol. Ag salt of a colloid or a mixt. of colloidal matter in proportions suitable for stopping the inhibition of the product by the surface while leaving it fluid at the ordinary temp. A soln. of acid citrate of Ag is mixed with a soln. of caseinate of soda and to the mixt. is added a suitable proportion of gelatin. A small quantity of resorcinol may also be added; it gives various tints to the printed positive.

Color photography. A. GLEICHMAR. Brit. 148,787, Jan. 29, 1920. To obtain the 3-component negatives, the blue and green negatives are combined together by a folding strip with their film faces in contact and exposed through a green filter which passes blue and some yellow but absorbs the deleterious red rays. The red negative is exposed directly through the transparent reflector. The red negative is then registered with the green negative and attached by a folding strip. The three negatives are opened out and the positives printed onto a single transparent foil previously creased for subsequent folding. The foil has sep. color areas, is printed through the back, and may be sensitized with silver bromide or dichromate. After developing and finishing the print the foil is folded to bring the prints in register and is mounted on paper or between glass plates when transparencies are required.

Color photography. F. M. WARNER. U. S. 1,358,802, Nov. 16. A positive transparency in natural colors is produced by making a negative of the subject on a sensitive plate having a color screen of different colored elements, making a monochrome positive from this screen negative on a plate coated with a panchromatic emulsion, and mounting the monochrome positive in registered position with a color screen similar to the screen through which the negative was taken.

Desensitizing photosensitive silver emulsions. N. SULZBERGER. U. S. 1,361,352, Dec. 7. Photo prints or films containing light-sensitive Ag compds. are desensitized or "fixed" by treating them with a soln. of Pd-NH_4 -protochloride.

Printing surfaces. E. SAUER. Brit. 151,405, July 15, 1919. A method of producing a printing-plate make-ready for printing consists in covering a sheet of more or less transparent material such as acetylcellulose, celluloid, paper, or casein, on both sides with sensitized gelatin, exposing it to light through a negative and developing in the usual manner. The relief thereby formed on the side adjacent to the negative forms the printing surface, and the relief on the opposite side forms the make-ready. In prep. the plate, a layer of gelatin is applied to a polished surface, the transparent sheet is laid upon the gelatin, and a second layer of gelatin applied, after which the plate is removed from the support and chromated before use.

6—INORGANIC CHEMISTRY

H. I. SCHLESINGER

Stereochemistry of the ruthenium atom. A. WERNER and A. P. SMIRNOFF. *Helvetica Chim. Acta* 3, 737-47(1920).—Some uncertainty remains as to the co-

ordination no. of Ru; in some compds. it appears to hold directly only 5 groups, e. g., $R_3[RuCl_5]$, and in some coordinatively satd. compds. the sixth group seems to be not entirely equiv. to the remaining 5. In attempts to clear up this uncertainty much difficulty was met in prepg. suitable starting material from halogen complexes of 3-4-valent Ru. It was finally found possible to replace NH_3 by en in $[(NO)(OH)Ru(NH_3)_4]X_5$. The new compds. of $[(NO)(OH)Ru en_3]$ are yellow to orange and yield neutral, pure yellow solns. With concd. halogen acid they yield $[(NO)H_2O Ru en_3]X_4$, orange, reacts acid, and this upon treatment with acid under pressure and at higher temp. yields $[(NO)X Ru en_3]X_3$, deep brown-red. The following salts of $[(NO)(OH)Ru en_3]$ are described: iodide, bromide, nitrate, chloride, sulfate, perchlorate, all anhydrous. It was unknown whether this series has the 1,2 or 1,6 conformation; attempts to obtain optical isomers were made with 5 different optically active acids; as all were unsuccessful, it is concluded that only the 1,6 form was present.

A. R. MIDDLETON

Complex compounds of thiocyanates and arsenious acid. FRITZ EPHRAIM. *Helvetica Chim. Acta* 3, 800-5(1920).—Upon treatment of a soln. of 39.6 g. As_2O_3 and 11.2 g. KOH in 50 cc. water with a soln. of 9.7 g. KSCN in 20 cc. water, the mixt. solidified to a paste with slight heat evolution. Upon recrystn. from hot water small hexagonal plates of compn. $KSCN.2As_2O_3$ were obtained; upon evapn. of the mother liquor better crystals were obtained which contained alkali and appeared to be mixed crystals containing water. Compns. approximating to $KSCN.2KOH.2As_2O_3$ and $KSCN.2KOH.3As_2O_3$ were obtained. They are but feebly complex and quite close to the "double salts." The physiological action of As in them is distinctly slower than that of an equiv. amt. of free arsenite.

A. R. M.

Solubility of iodine in hydriodic, hydrobromic and hydrochloric acids and the formation of the corresponding polyhalogenated acids. I. E. OLIVERI-MANDALÀ AND A. ANGENICA. Univ. Palermo. *Gazz. chim. ital.* 50, I, 273-81(1920).—It is known that the halogens are more sol. in solns. of the hydracids than in pure H_2O . This paper is a portion of a systematic study of this subject and relates to the soly. of I in HI, HBr and HCl. The expts. were done by agitating I in solns. of the hydracids of known concn. in a thermostat at 25°. The I in soln. was detd. with standard As_2O_3 solns. The results are given in a series of tables and show that the ratio of I/HI, I/HBr and I/HCl is a function of the concn. and increases slightly with increase in the concn. of the halogen acids. The same behavior was shown by I in KI soln. The soly. of I in HI and in KI is the same for the same concns. This same fact was observed by comparing the soly. of I in HCl and HBr and in KCl and KBr, resp. The increase in soly. of I in hydracids may be explained by the formation of polyhalogen acids, $HI.I_2$, $HCl.I_2$ and $HBr.I_2$, analogous to the corresponding alk. salts. The existence of salts like $KI.I_2$, $KCl.I_2$ and $CsBr.12I_2$ has been studied and established in various ways O.-M. and A. have used the cryoscopic method on the HI solns. of I and found that the f. p. of the HI soln. remains unchanged on the addition of the I. Moreover the disocn. of the HI at concn. 0.1 to 0.3 N is equal to that of HI_3 . The results in general indicate that the soly. of I in HI, HBr and HCl and in the corresponding alk. salts is sp. for the halogen ion and is independent of the positive ion. E. J. WITZEMANN

Aluminates of sodium. Equilibria in the system $Na_2O-Al_2O_3-H_2O$. F. GOUVERIAAN. *Proc. Acad. Sci. Amsterdam* 23, 120-42(1920); cf. C. A. 14, 664.—The 30° isotherm was detd. As solid phase 3 different forms of $Al(OH)_3$ were used: (a) was pptd. by NH_4OH and dried at 130-40° until the H_2O content approximated the formula. The particles swelled little or none even after long shaking with water, but very rapidly with NaOH stronger than 2 N. (b) was formed by very gradual decompn. of aluminate soln. by atm. CO_2 ; it adsorbed ions to less extent than (a); magnified 400-fold it showed no cryst. structure; it swells with concd. NaOH but not with dil. (c) is a

new finely crystd. form obtained by shaking (a) with 0.5–2.0 N NaOH. No swelling was observed, but a gradual transition of the irregularly shaped, various-sized gelatinous particles to bar-shaped crystals quite uniform in size and shape took place, complete transition requiring a few months. Analysis of the crystals, dried at 100–10°, showed $Al_2O_3 \cdot 3H_2O$. These 3 forms, upon heating at 300–400°, gave the forms of Al_2O_3 (d), (e) and (f); (d) and (e) had still the appearance of a shriveled, gelatinous mass and were still capable of swelling or crystg. in NaOH according to the concn.; (f) was no longer distinctly cryst. The gel forms were much more sol. in NaOH and there is probably a continuous transition between these and the cryst. hydrate. The gel hydroxide must be considered a metastable phase of variable compn. It adsorbs variable quantities of alkali. Two stable aluminates were found, $4Na_2O \cdot 3Al_2O_3 \cdot 16H_2O$, diamond-shaped crystals, and $4Na_2O \cdot Al_2O_3 \cdot 10H_2O$, needle-shaped crystals, very hygroscopic. Both form strongly incongruent satd. solns. At 30° Al_2O_3 is metastable as regards the hydrate.

A. R. MIDDLETON

Monoclinic double selenates of the copper group. A. E. H. TURTON. *Proc. Roy. Soc. (London)* 98A, 67–95 (1920).—The K, Rb, Cs and NH_4 salts of the series $R_2Cu(SelO_4)_2 \cdot 6H_2O$ were prepd. in pure condition. Their complete crystallographic description is given. The general results confirm those previously obtained for the Mg, Zn, Fe, Ni and Co groups and for the analogous double sulfates. The absolute values of the constants expressing the morphological properties are considerably different from those of the other groups (in this resembling the Cu double sulfates) but the relationships are remarkably similar. The same progression with at. wt. of the alkali metal and the same similarity of the NH_4 to the Rb salt are apparent. A. R. M.

The zincates of sodium. Equilibrium in the system $Na_2O-ZnO-H_2O$. F. GOUVERLAAN. *Univ. technique, Delft. Rec. trav. chim.* 39, 505–14 (1920); cf. C. A. 14, 664.—The data in the literature on the reactions between the strong sol. bases of the light metals (KOH, NaOH) and the weak insol. hydroxides of the heavy metals are contradictory and indefinite. At present the compds. formed in these reactions and the conditions under which they are stable are little known. Even some of the hydroxides themselves are unknown. In this paper the compds. formed on dissolving $Zn(OH)_2$ in NaOH were detd. and the conditions under which they are stable were established. The soly. diagram for the system $Na_2O-ZnO-H_2O$ at a const. temp. was detd. The ZnO used was obtained by pptg. $Zn(NO_3)_2$ with NH_4OH . The ppt. was washed completely with boiling H_2O and then dried at 140–50°. It was also obtained by heating $ZnCO_3$. All the measurements were made at 30° and the results are given in a table and a diagram. They show that at 30° there is only one stable Na zincate (a) with the compn. $Na_2O \cdot ZnO \cdot 4H_2O$. All the other zincates mentioned in the literature are not stable, or else do not exist at all. G. found from the diagram that a will undergo decompn. on adding H_2O with the sepn. of ZnO . Dil. solns. of NaOH have the same effect. The course of the isotherm shows that ZnO seps. with NaOH solns. having less than 1 g. NaOH per 2 g. H_2O when added to solid a. Accordingly, in order to obtain crystals of a free from ZnO , this high concn. of NaOH is necessary. G. repeated the prepn. of a according to Comey and Jackson (*Am. Chem. J.* 11, 145 (1889)) and obtained a pure product. It was observed that when washed with 96% EtOH this prepn. is slowly converted into ZnO or $Zn(OH)_2$ which probably accounts for the products of improbable compn. obtained by them. $Zn(OH)_2$ pptd. with NH_4OH and washed with hot H_2O and dried at 100° contained only 0.6% H_2O showing that $Zn(OH)_2$ is not stable. $Zn(OH)_2$ pptd. from $ZnCl_2$ and washed for 48 hrs. still contained 0.30% Cl after drying at 100°. The gelatinous $Zn(OH)_2$ is more sol. in NaOH soln. than ZnO and on standing its soly. diminishes which shows that the former is metastable with respect to ZnO and that the phases are in process of conversion into ZnO . Gelatinous $Zn(OH)_2$ should be considered as phases with a variable H_2O content. Klein (C. A. 6, 1411) describes

3 forms of $\text{Zn}(\text{OH})_2$. The cryst. form was obtained by G. thus: 50 cc. of 0.1 N KOH were added drop by drop to a 0.1 N soln. of ZnSO_4 . At first the $\text{Zn}(\text{OH})_2$ formed dissolved but presently it remained turbid. On agitating strongly and adding if possible a crystal of $\text{Zn}(\text{OH})_2$ a heavy and sandy ppt. sepd. which was microscopically homogeneous and when dried at 40–50° gave on analysis results corresponding to $\text{Zn}(\text{OH})_2$. The same result was obtained with KOH solns. varying from 4.0 to 0.1 N in concn. When this cryst. $\text{Zn}(\text{OH})_2$ was used in the studies of the ternary system it was found that at 30° it is metastable with respect to ZnO . The cryst. $\text{Zn}(\text{OH})_2$ is more sol. in NaOH soln. than ZnO . E. J. WITZEMANN

The arsenites of lithium and potassium. F. A. H. SCHREINEMAKERS AND MISS W. C. DE BAAT. Univ. Leyden. *Rec. trav. chim.* 39, 423–8 (1920).—In previous papers the authors have studied the combinations of As_2O_3 with salts (C. A. 11, 2865), the arsenites of NH_4 (*Verst. Kon. Akad. Wetensch. Amsterdam*, Feb. 1915; cf. C. A. 9, 1566) and of Na (C. A. 11, 2174) and now report on those of Li and K. In order to find the Li arsenites that exist at 25° the ternary system $\text{Li}_2\text{O}-\text{As}_2\text{O}_3-\text{H}_2\text{O}$ was studied. The only salt found was LiAsO_3 . In order to obtain this equil. the solns. were agitated day and night for 4 to 6 weeks in the thermostat. The data and diagram for this ternary system are given in full. The similar system with H_2O was also studied and the ternary diagram was detd. Two K salts were found: $\text{K}_2\text{As}_2\text{O}_7$ and $\text{K}_4\text{As}_6\text{O}_{12}\cdot 12\text{H}_2\text{O}$. Besides the 3 arsenites already given NH_4AsO_3 , NaAsO_3 , $\text{Na}_4\text{As}_2\text{O}_6$, $\text{Na}_3\text{As}_3\text{O}_{11}\cdot 26\text{H}_2\text{O}$ and $\text{Na}_4\text{As}_5\text{O}_{14}\cdot 9\text{H}_2\text{O}$ were previously obtained. By analogy with other acids these salts ought to be derived from the acids HAsO_3 , $\text{H}_4\text{As}_2\text{O}_7$ and H_3AsO_3 . Of these no salts of H_3AsO_3 were found and 3 of the salts given are not directly derivable from any of these acids. Ways of deriving these salts from these simple acids are described. E. J. WITZEMANN

Preparation of selenium oxychloride. VICTOR LENHER. *J. Am. Chem. Soc.* 42, 2498–2500 (1920).—Three methods are given: (1) direct union of SeO_2 and SeCl_4 in CCl_4 or CHCl_3 ; SeCl_4 is prepd. by chlorinating Se suspended in CCl_4 , the Se_2Cl_2 which is first formed being a good solvent for Se. This process is useful in sepg. Se from elements the chlorides of which are insol. in CCl_4 . (2) Partial hydrolysis of SeCl_4 . (3) Dehydration of $\text{SeO}_3\cdot 2\text{HCl}$ formed by contact at moderately low temp. of HCl gas and SeO_2 . The amber liquid can be mixed with P_2O_5 or CaCl_2 and distd. A. R. M.

Hydrolysis of zirconyl chloride and sulfate at zero and twenty degrees. F. P. VENABLE AND D. H. JACKSON. *J. Am. Chem. Soc.* 42, 2531–4 (1920).—Hydrolysis was measured both by relative cond. and by pptn. with HIO_4 at 3 different dilns. Both methods indicated an initial temp. adjustment on dissolving the salts followed by decrease of resistance; equil. or a state of very slow change was reached in 2–3 hrs. A. R. MIDDLETON

Hydrolysis of the silicates of sodium. R. H. BOGUE. *J. Am. Chem. Soc.* 42, 2575–82 (1920).—H-ion concns. were measured at 30° of solns. $1/2$, 0.1, 0.05, 0.02 and 0.01 molar of 7 silicates in which $\text{SiO}_2/\text{Na}_2\text{O}$ varied from 4/1 to 1/1. The data indicate either that these silicates are much less hydrolyzed in dil. soln. (1.58–28.43% in 0.01 molar soln.) than has been supposed or else that the electrometric method is unsuitable for measuring such dissoen., possibly because colloidal SiO_2 is able to adsorb or retard OH ion. The data confirm Na_2SiO_3 to be most hydrolyzed and the degree of hydrolysis to decrease as more SiO_2 is added to the mol. A. R. MIDDLETON

Action of ozone on alkali metals, ammonia and substitution products of ammonia. W. STRUCKER AND H. THIENEMANN. *Ber.* 53, 2096–2113 (1920); cf. Manchot, C. A. 2, 952; 7, 2358.—In order to study the action of O_3 on alk. metals these were dissolved in liquid NH_3 . O_3 formed first a gelatinous white mass, later found to be hydroxide, the blue color vanished, then the ppt. dissolved and a deep orange liquid and ppt. was formed which showed all the reactions described by Manchot. Readiness of forma-

tion and stability increased in the order Na, K, Rb, Cs. Sepia brown ozonides of Ba and Ca were also obtained which decompd. completely as soon as NH_3 was evapd. The products from alk. metals contained much nitrate; study of the action of O_3 on pure liquid NH_3 showed that the reactions $2\text{NH}_3 + 4\text{O}_3 = \text{NH}_4\text{NO}_3 + 4\text{O}_2 + \text{H}_2\text{O}$ and $2\text{NH}_3 + 3\text{O}_3 = \text{NH}_4\text{NO}_2 + 3\text{O}_2 + \text{H}_2\text{O}$ take place; the residues contained about 98% nitrate and 2% nitrite. First action of O_3 formed an intense yellow color, indicating that an unstable ozonide is formed which decomposes into nitrate and nitrite. With aq. solns. of NH_3 O_3 was found to react less completely as diln. increased. Absence of moisture did not prevent reaction, but it was slow at first and increased rapidly, indicating that water exerts an accelerating effect. Owing to instability of the ozonides satisfactory data upon their compn. could not be obtained. With K and Rb 2/3 to 3/4 at. of O was found per mol. of hydroxide and nearly 1 at. for Cs. NH_4OH with O_3 formed only $\text{NH}_4\text{OH} \cdot \text{HN}\text{O}_2$; $\text{N}_2\text{H}_4\text{OH}$ formed chiefly N_2 and H_2O with a little NH_4NO_3 and $\text{N}_2\text{H}_4\text{NO}_3$. MeNH_2 reacted more violently than NH_3 with formation of HCHO , and nitrate and nitrite of NH_4 and of the amine. Me_2NH formed HCHO , nitrate, nitrite, HCO_2H and also AcOH . Explosion followed as soon as O_3 was led into Me_2N at -80° ; a 5–10% soln. in CCl_4 , or better, CHCl_3 gave NH_4Cl and the hydrochloride of $\text{Me}_2\text{N} \cdot \text{O}$. Other amines reacted similarly. A. R. MIDDLETON

Preliminary note on the action of fluorine on potassium bisulfate. E. BRUNNER. *Helvetica Chim. Acta* 3, 818–24 (1920).— F_2 was delivered for 1 hr., slightly above the surface of a cold satd. soln. of KHSO_4 with frequent shaking of the flask. The product showed the following reactions: strong odor of O_3 which vanished after 3 days; 0.1 N KMnO_4 was not decolorized after 8 hrs.; strong sepn. of I_2 from KI ; immediate black ppt. from alk. MnCl_2 soln.; the last two reactions persisted long after the odor of O_3 had ceased. From this it is concluded that the reactions depended not on O_3 or H_2O_2 but on an oxidation product of H_2SO_4 . An appended note upon statements of Treadwell (Quant. Anal., 5th ed., p. 682) concerning Hempel's gas volumetric method for F, presents evidence that pressure changes are without effect upon the accuracy and that the white sublimate noted by T. is due to presence of moisture admitted under reduced pressures. The method cannot be used for fluorides which are difficultly decomposed. A specimen of fused PbF_2 was indicated as 65% PbF_2 . After heating 8 hrs. with 2 N NaOH , neutralizing with H_2SO_4 and removing PbSO_4 , the residual Na salt gave F corresponding to 89.6% PbF_2 . A. R. MIDDLETON

Preparation and physical properties of carbonyl chloride. R. H. ATKINSON, C. T. HEYCOCK AND W. J. POPE. *J. Chem. Soc.* 117, 1410–26 (1920).—In the works prepn. of COCl_2 by the Paternò process (*Gazz. chim. ital.* 8, 233 (1978)) difficulties were experienced in finding charcoals effective as catalysts. The following proved exceptionally effective: fresh ox bones crushed and embedded in sand were calcined in a muffle furnace, extd. with hot HCl , then with water and heated again in sand; the bone charcoal was kept at red heat for some time in a current of dry Cl_2 . With 10 g. of this 10 kg. of COCl_2 were obtained without indication of loss of activity. COCl_2 was formed freely at room temp. when Cl_2 and a somewhat greater vol. of CO was passed over the charcoal; at $40\text{--}50^\circ$ combination was more rapid than the mixed gases could be supplied. Even more efficient proved the highly activated wood charcoal used in army respirators. At 14° more than 20 kg. of COCl_2 were obtained with 10 g. without loss of activity. Expts. proved that with CO containing H_2 no HCl was formed below 70° ; a little was formed at 80° and much more at 90° . The COCl_2 was free from Cl_2 when reaction took place within the range $50\text{--}200^\circ$. The dissocn. curve of COCl_2 was detd. The dissocn. was found to be about 1% at temps. slightly above 200° and to increase to 55–6% at 503° . Heat of formation, calcd. from degree of dissocn. is $+25.4^\circ\text{C. at } 475^\circ$ and $+23.4^\circ\text{C. at } 425^\circ$. The vapor pressure curve was detd. between -100° and $+100^\circ$. The curve begins to fall rapidly only below -40° and considerable losses

will occur in prepn. unless effluent gases are cooled below this temp. The soly. of COCl_2 in various org. solvents was detd.; toluene, coal-tar xylene and chlorobenzene were by far the best of those examd. The temp. of melting COCl_2 remained const. at -126° and on very slow cooling crystals first appeared at -123° . Densities, 1.679 at -104° ; 1.434 at 0° ; 1.314 at $+49.9^\circ$. Mean coeff. of cubical expansion between -79° and $+50^\circ = 1.77 \times 10^{-3}$.

A. R. MIDDLETON

The system cupric oxide, cuprous oxide, oxygen. F. H. SMYTH AND H. S. ROBERTS. *J. Am. Chem. Soc.* **42**, 2582-2607(1920).—Solid soln. of Cu_2O in CuO does not take place in the temp. range where both oxides remain solid. The pressure-temp. equil. curves have been established over the range in which the oxides remain solid, below the eutectic pt., and above the eutectic pt. where CuO remains the solid phase, up to 1233° . The quadruple point lies at 1080.2° and 390 mm. Pure CuO does not melt without dissocn. below 1233° . The general direction of the equil. curve is indicated for the system when Cu_2O remains the only solid phase and the theoretical deduction is confirmed that the equil. pressure drops with rise of temp.

A. R. MIDDLETON

The properties of pure hydrogen peroxide (MAASS, HATCHER) 2. The chemistry and crystallography of some fluorides of cobalt, nickel, manganese and copper (EDMISTER, COOPER) 2.

KNOEVENAGEL, E.: *Praktikum des anorganischen Chemikers*. Einführung in die anorganische Chemie auf experimenteller Grundlage. 3rd Ed. Berlin: Walter de Gruyter & Co. M. 45.

MOLDENHAUER, WILHELM: *Die Reaktionen des freien Stickstoffs*. Berlin: Gehr. Bornträger. 178 pp. M. 26. For review see *Z. angew. Chem.* **33**, I, 320(1920).

SCHWARZ, R.: *Chemie der anorganischen Komplexverbindungen*. Ein Grundriss für Studierende. Berlin: Walter de Gruyter & Co. M. 10.

7—ANALYTICAL CHEMISTRY

WILLIAM T. HALL

The position of analytical chemistry in France. L. COPMAN. *Analyst* **45**, 440-4 (1920).—A discussion of the professional side of the subject.

E. J. C.

Progress in the field of metal analysis in 1919. TH. DÖRING. *Chem.-Ztg.* **44**, 893-4, 933-5, 961-3, 967-9(1920).—Numerous references accompany this review.

Cf. *C. A.* **14**, 504.

E. H.

Unit weights for the purchase of reagents. W. D. COLLINS. *J. Ind. Eng. Chem.* **12**, 1206(1920).—A note urging the use of metric units in purchasing reagents. A list of reagents is given with suggested unit wts.

F. W. SMITHER

Electroanalytical practice. W. BÖTTGER. Leipzig. *Z. Elektrochem.* **26**, 445-52 (1920).—The electrodeposition of Hg from nitrate solns. (HgNO_3 or $\text{Hg}(\text{NO}_3)_2$) + HNO_3 and from chloride soln. + HNO_3 or + KCN can be carried out quant. The causes for deviations between the wt. of metal deposited and the theoretical amt. are discussed and the magnitude of these deviations is detd. In drying the electrode, with its deposit the H_2O may be taken up with purified alc. and Et_2O provided the electrode afterwards is exposed to the air for a short time (10-15 min.). The loss which occurs when the electrode is kept in a desiccator is very small. The deviation between the theoretical and the actual wt. of metal deposited in an acid soln. (nitrate soln. + 1 cc. HNO_3 , d. 1.4, with 1.4 to 1.5 volts, or chloride soln. + 1 cc. HNO_3 with 1.8 to 3.3 v.) with a silvered gauze electrode amounted to 0.4 mg., while from a cyanide soln. it amounted to 0.6 mg. The accuracy of the cyanide method decreases with increase in the amt. of KCN and with the current strength.

H. JERMAIN CREIGHTON

A simplified method for the volumetric determination of acids and bases that form insoluble or very difficultly soluble salts. HANS TH. BUCHERER. *Z. anal. Chem.* 59, 297-302(1920).—A modification of Wildenstein's method (cf. *Z. anal. Chem.* 1, 432). *Detn. of H_2SO_4 by means of $BaCl_2$ or vice versa:* Dil. the sol. to be examd. (containing the equiv. of about 1 g. H_2SO_4) to 500 cc. with hot H_2O , and add N $BaCl_2$ soln. until it can be no longer noted with certainty whether $BaSO_4$ forms on further addition of the precipitant. Filter off about 5-10 cc., test half with a drop of $BaCl_2$ soln. and the other half with a drop of dil. H_2SO_4 . If the test shows that all of the H_2SO_4 has not been pptd., carefully add 0.5-1 cc. N $BaCl_2$ soln., filter and test as before. By this means the amt. of $BaCl_2$ required can be detd. within 0.1-0.2 cc. Now repeat the detn. on a new portion of the original soln. adding within 0.2-0.3 cc. the amt. of N $BaCl_2$ soln. used in the preliminary detn. Complete the detn. with 0.1 N $BaCl_2$ soln., filtering and testing as above. The method can be used for detg: Ca or $H_2C_2O_4$ as CaC_2O_4 , Mg and P_2O_5 as NH_4MgPO_4 , or Ca and Mg in the same soln. without filtering off the CaC_2O_4 .
F. W. SMITHER

Gas analysis by absorption and titration. R. S. TOUR. *Chem. Met. Eng.* 23, 1104-6(1920).—For the more accurate detn. of NH_3 , CO_2 , SO_2 , H_2S , etc., in gases, it is best to absorb the constituent in a suitable reagent, measure the vol. of residual gas, and titrate the absorbing soln. The measurement of the gas and its vol. correction is greatly simplified by the use of a compensometer, and the final calcn. may be accomplished graphically with the aid of a nomograph. The compensometer described may be used to measure a gas at room temp. and pressure and at the same time to give a factor for converting this vol. to standard conditions or to read directly the corrected standard vol. It is designed for an accuracy of 0.5%. The gas to be measured is drawn through a double-inlet stopcock displacing the H_2O in the gas flask to a fixed mark. The stopcock is then turned to communicate with a Hg manometer the other leg of which connects with the compensometer tube, which in turn is completely enclosed by the gas flask. The compensometer pressure on one leg of the manometer indicates the temp. correction, and if a drop of H_2O is inclosed within the compensometer, the correction includes that for H_2O vapor pressure. The pressure of the gas in the flask acting on the other leg of the manometer tends to balance the compensometer pressure and the manometer actually indicates the net correction to be applied. Formulas are given for graduating the manometric scale, and a nomograph for simplifying the calculations is included.
S. G. SIMPSON

The determination of oxygen in iron. II. P. OBERHOFFER AND O. V. KEIL. *Stahl u. Eisen* 40, 812-4(1920).—The app. already described (*C. A.* 13, 294) has been simplified so that better results are obtained in a shorter time. Instead of the Hg pump for evacuating the app. a common water pump is used and the total space to be evacuated is reduced to 150 cc. By 3 evacuations with intermediate filling with H, the O in the app. is reduced to 0.00006 g. After one analysis is completed the furnace is withdrawn and the tube only allowed to cool to 500°. It has been found possible to introduce a new sample without perceptible oxidation at this temp. About 1 hr. is required for an entire analysis. The app. appears to be the simplest and best that has been devised for this detn.
W. T. H.

The determination of sulfur in cast iron, steel, ores, slags and fuels by combustion in oxygen. A. VITA. *Stahl u. Eisen* 40, 933-8(1920).—The sample is heated in a current of O exactly as in the detn. of C using a temp. of 1200° in the case of Fe and steel and 1350° for slags and fuels. When substances like $CaSO_4$ are present about 45 min. are required. S present as $BaSO_4$ and as alkali sulfate is not obtained. If free S is given off during the heating the products of combustion must be passed through a heated capillary tube. The combustion gases are absorbed in an aqueous soln. of KI and KIO_3 , and each mol. of SO_2 or of SO_3 liberates 3 mols. of I_2 . With low S, the analysis

is finished colorimetrically, using K_2CrO_4 soln. for comparison. For quantities of S of about 0.01%, about 0.5 g. of substance should be used. With larger quantities of S it is better to det. the liberated I by titration with $Na_2S_2O_4$. The results obtained with various kinds of Fe and steel as well as with coal, coke and sulfide ores, slags, etc., indicate that the results are accurate. By taking special precautions with regard to the pressure of the gas and the concn. of the absorbent, it was found possible to det. C and S simultaneously by the combustion of a single sample. An interesting discussion followed the reading of the paper.

W. T. HALL

Determination of vanadium in steel. ÉMILE JABOULAY. *Rev. métal.* 17, 627-9 (1920).—This method may be carried out in the soln. in which Cr is detd. volumetrically with $FeSO_4$ and $KMnO_4$. It is based upon the fact that HVO_3 can be reduced with $FeSO_4$ and the excess of the latter may be destroyed with $K_2Cr_2O_7$ which in small amts. has no effect upon the subsequent titration of the divanadyl salt with $KMnO_4$. Dissolve 1 g. of the steel in dil. H_2SO_4 , heating to hasten soln. Add HNO_3 drop by drop until effervescence stops and the Fe has been oxidized. Boil gently and add satd. $KMnO_4$ very slowly until a slight ppt. of MnO_2 persists. Dilute to 200 cc. Boil 10 min. and filter MnO_2 and any WO_3 . If Cr is present it may be detd. in the regular way by adding excess $FeSO_4$ and titrating back with $KMnO_4$. Cool the soln. and add $FeSO_4$ in slight excess, using $K_3Fe(CN)_6$ as an outside indicator. Dilute the soln. to about 700 cc. Add 1 cc. of 0.1 N $K_2Cr_2O_7$ to oxidize excess $FeSO_4$ and at once titrate rapidly with 0.1 N $KMnO_4$. The presence of Ni may cause high results due to the green color of its salts.

S. G. SIMPSON

Critical investigation of methods for the determination of phosphorus in iron, steel, ores and slags. HUGO KINDER. *Stahl u. Eisen* 40, 381-7, 468-73 (1920).—Previous work of the commission of German metallurgical chemists has indicated the advisability of weighing the ammonium phosphomolybdate ppt., drying at 105° (Finkener) or at 450° (Meinecke). In the first case the ppt. contains 1.637% P and in the second case 1.722% P. The presence of Cu, Ni, Co, Cr and sulfate was found not to affect the result appreciably. W and Ti cause pptn. of P in the acid-insol. residue. Mo causes slight errors and with V a complex is formed. As causes high results unless it is removed by evapn. with HBr and HCl. By reducing the V to the quadrivalent condition just before treatment with molybdate, this element causes no trouble. To show the accuracy of the method a number of ores and samples of Fe and steel were analyzed carefully and the results tabulated. With the ores, only the gravimetric method was tried, but with iron and steel the results were compared with those obtained by alkalimetric titration of the yellow ppt. The methods used are described in detail and the paper shows the accuracy that can be obtained in this class of work. W. T. H.

Volumetric determination of iron, of mercury and of vanadium in two states of oxidation in the same liquid. M. G. HINARD. *Ann. chim. anal. chim. appl.* 2, 297-9 (1920).—This method is recommended for rapidity rather than for great precision. Two cases require different modifications in the procedure: (1) where the 3 metals are all in a state of maximum oxidation, and (2) where they are in different states of oxidation. (1) Ppt. $HgS + S$ with H_2S , let settle, filter warm, wash with H_2O , and save the filtrate. Dissolve the ppt. with Br water, boil off Br, and filter off any residual S. Make this filtrate alk. with KOH, and add a known vol. of standard KCN soln. with KI as indicator. Titrate excess KCN with $AgNO_3$ and calc. Hg. To det. Fe^{+++} boil off H_2S from the original filtrate, oxidize with Br water and ppt. at 100° with KOH. Redissolve in HCl and reppt. with KOH. Dissolve the ppt. in hot H_2SO_4 , cool, add known excess of KI soln., titrate free I with $Na_2S_2O_3$ and calc. Fe. To det. V, add to a measured vol. of the original soln. enough KOH or NH_4OH to form a ppt. Redissolve this with slight excess of H_2SO_4 and continue as for Fe, but this time add $Na_2S_2O_3$ until the soln. changes from yellow to blue. By sufficient diln. the change is always sharp. With

Fe known, V is detd. by difference. (2) Test first for Fe by $K_2Fe(CN)_6$. A considerable amt. of Hg is detrimental to this test, so if Hg is present in large amt. remove by addition of concd. NaCl soln. and rapid filtration. With only Fe and V present, their detn. can be made as above by oxidation with Br water. With Hg^{++} present, treat a sep. portion of soln. with concd. NaCl soln., cool as low as practicable, filter, wash, and det. Hg^{++} in the filtrate and thus by difference the Hg^+ . In absence of Fe^{++} , det. Fe^{+++} and total V in the usual way. Then det. V_2O_5 by adding Br water followed by the regular excess KI and $Na_2S_2O_3$ method, deducting the vol. of $Na_2S_2O_3$ corresponding to Fe detd. previously. The $Na_2S_2O_3$ corresponding to V_2O_5 is then known and the V in the lower state of oxidation can be calcd. by difference. In presence of Fe^{++} , det. total Fe and V. Add to the liquid, not oxidized by Br, excess KI soln., and titrate for I. This corresponds exclusively to Fe^{+++} salt. Fe^{++} can then be detd. by difference with V and Fe^{+++} known.

C. C. DAVIS

Gold analyses. PAUL DRAWE. *Z. angew. Chem.* 33, I, 272(1920).—Instead of attempting to ext. the other metals by treatment with HNO_3 and calling the insol. residue Au, the following procedure is recommended. Dissolve 0.5 g. of metal in 50 cc. aqua regia, evap. to dryness and treat the residue with dil. HCl. Filter off AgCl and ppt. Cu in the filtrate with KOH. Finally ppt. the Au with H_2O_2 in slightly alk. soln.

W. T. H.

Gold analyses. H. WOLFF AND N. SINGALOWSKY. *Z. angew. Chem.* 33, I, 298 (1920).—Data are given which show, in disagreement with the assumption of Drawe (see preceding abstract) that the results obtained in parting Au-Ag buttons with HNO_3 in the orthodox manner are remarkably accurate. Starting with pure Au and pure Ag, accurate results were obtained in parting a button and the test for Ag in the residual Au was negative.

W. T. H.

Separation of mercury from other elements by distillation from hydrochloric acid solution. W. STRECKER AND KARL CONRADT. *Ber.* 53B, 2113-27(1920).—Expts. are detailed which had for their objective fixing conditions under which Hg might be completely volatilized as chloride. A temp. of $150-60^\circ$ was found sufficient and this was satisfactorily obtained with either H_2SO_4 or H_3PO_4 . A current of HCl gas was proved not essential; dropping a 9:1 concd. HCl-HBr mixt. into the flask resulted in complete removal of Hg from the distn. flask in 1 hr. $HgCl_2$, but not $HgBr_2$, could be completely distd. from H_3PO_4 , but not from H_2SO_4 , by dropping about 200 cc. H_2O during distn. Necessity for addition of HCl with H_2SO_4 is considered due to formation of the less volatile sulfate, which must be decompd.; the acceleration effect of HBr may be due to greater stability by $HgBr_2$ toward the action of H_2SO_4 . $HgBr_2$ distills over slowly at 150° but much more rapidly at 160° . Sepns. of Hg from Cu, Pb, Cd, Fe and Ba were made.

A. R. MIDDLETON

Gravimetric methods of analysis. XIV. L. W. WINKLER. *Z. angew. Chem.* 33, I, 287-8(1920).—XVII. *Detn. of H_2SO_4 in the presence of Ca:* No $CaSO_4$ is pptd. with the $BaSO_4$ if the precipitation takes place in the presence of hot 10% HCl, but it is necessary to apply a correction factor because of incomplete pptn. XVIII. *Detn. of H_2SO_4 in the presence of H_3PO_4 :* In this case the values are too high when the pptn. is made in hot 10% HCl soln. and a negative correction must be applied. XIX. *Detn. of H_2SO_4 in the presence of Cr:* In this case the precipitation of $BaSO_4$ is very incomplete, but the sulfate in the filtrate can be converted into $BaSO_4$ by evapg. the filtrate to dryness in the presence of excess HCl, moistening the residue with HCl and dilg. with water. XV. *Ibid* 299.—XX. *Detn. of Ba as $BaSO_4$:* To 100 cc. of soln., containing 0.20-0.1 g. Ba, add 1.0 g. of NH_4Cl , 1 cc. of N HCl and a small piece of Cd foil. Heat to boiling and add 5 cc. of 5% $(NH_4)_2SO_4$ soln. Filter off the $BaSO_4$ ppt. after standing overnight. With 300 mg. of ignited ppt., the results are about 0.3 mg. too high, but with smaller quantities of Ba usually the values are a trifle low. W. T. H.

Determination of phosphoric acid in phosphates of the heavy metals. F. SEBLIG-MANN. *Berlin. Chem.-Ztg.* **44**, 599(1920).—In technical practice phosphates are commonly estd. by titration with uranyl acetate. The alkali phosphates are taken up with dil. HNO_3 , neutralized with NH_4OH , slightly acidified with AcOH and titrated. The phosphates of the alkaline earths are dissolved in concd. aq. alkali and dild. to vol. and an aliquot part is acidified and titrated. The following procedure is recommended for phosphates of the heavy metals: Stir 10 g. very finely pulverized sample into 12 g. NaOH (d. 1.38); warm, shaking continually, to $90-5^\circ$. Soln. is effected in 15 min. Dil. with three or four times the quantity of water, heat, and then dil. to 1 l. Filter off an aliquot portion, make slightly acid with AcOH , and titrate as usual, with uranyl acetate. The analysis takes 1 hr. and gives good technical results.

ALBERT SALATHE

Volumetric determination of hydrosulfide, sulfide, thiosulfate and sulfite in the presence of one another. A. WÖBER. *Chem.-Ztg.* **44**, 601(1920); cf. Sander, *C. A.* **8**, 866, 1140; **9**, 2042, 2357.—I. In an aliquot part of the original soln. det. the $\text{SH}^- + \text{S}_2\text{O}_3^{--}$ as follows: Treat with a liberal excess of satd. HgCl_2 soln. These reactions take place: $2\text{S}^{--} + 3\text{HgCl}_2 \longrightarrow \text{Hg}_3\text{S}_2\text{Cl}_2 + 4\text{Cl}^-$, $\text{SO}_3^{--} + \text{HgCl}_2 \longrightarrow \text{Cl}^- + \text{HgClSO}_3$, $2\text{SH}^- + 3\text{HgCl}_2 \longrightarrow \text{Hg}_3\text{S}_2\text{Cl}_2 + 2\text{H}^+ + 4\text{Cl}^-$, $\text{S}_2\text{O}_3^{--} + 3\text{HgCl}_2 + 2\text{H}_2\text{O} \longrightarrow 4\text{H}^+ + \text{Hg}_3\text{S}_2\text{Cl}_2 + 2\text{SO}_4^{--} + 2\text{Cl}^-$. Add NH_4Cl to prevent pptn. of HgO and titrate the acidity with 0.1 N NaOH , using methyl orange. II. In another aliquot, ppt. the S^{--} and SH^- by treatment with CdCO_3 or ZnCO_3 , filter and in the filtrate decompose the bicarbonate formed by the reaction $\text{ZnCO}_3 + \text{SH}^- \longrightarrow \text{ZnS} + \text{HCO}_3^-$ by the careful addition of 0.1 N HCl . Add HgCl_2 and NH_4Cl and titrate this time the acidity due to $\text{S}_2\text{O}_3^{--}$ alone. III. Allow another aliquot to run into a known vol. of 0.1 N I_2 to which 5 or 10 cc. of 0.1 N HCl has been added. Titrate the excess of I_2 with standard $\text{Na}_2\text{S}_2\text{O}_3$ soln. With I_2 , the following reactions take place: $\text{S}^{--} + \text{I}_2 \longrightarrow \text{S} + 2\text{I}^-$, $\text{SH}^- + \text{I}_2 \longrightarrow 2\text{I}^- + \text{H}^+ + \text{S}$, $\text{SO}_3^{--} + \text{I}_2 + \text{H}_2\text{O} \longrightarrow \text{SO}_4^{--} + 2\text{I}^- + 2\text{H}^+$, $2\text{S}_2\text{O}_3^{--} + \text{I}_2 \longrightarrow \text{S}_4\text{O}_6^{--} + 2\text{I}^-$. Thus the total content of S compds. is measured. After the titration with thiosulfate, add methyl orange and titrate the acidity formed by the reaction between SO_3^{--} and SH^- with 0.1 N NaOH .

A. J. SALATHE

The chemical examination of antimony sulfides. A. VAN ROSSEM and P. DEEKER. *India Rubber J.* **60**, 905(1920).—After a study of the literature on the subject, the conclusion is drawn that the detn. of free S in Sb sulfides by extn. methods with org. solvents is impracticable, as Sb_2S_3 is decompd. by these solvents. In the absence of trisulfide, Sb_2S_4 is detd. by decomp. with HCl and measuring the H_2S liberated. $\text{Sb}_2\text{S}_3 + 6\text{HCl} \longrightarrow 2\text{SbCl}_3 + 3\text{H}_2\text{S} + 2\text{S}$. The S in the residue from the acid treatment minus the S formed by the reaction shown, gives the free S in the pentasulfide. Weber's method of free S detn. by strong NH_3 soln. of Sb_2S_3 was shown to give varying results. In barely ammoniacal solns., fairly accurate results were obtained. The methods of Weber and Sweet and of Repony for detg. moisture and water of crystn. were revized.

A. H. SMITH

Determination of sulfide sulfur in roasted blend. MAURICE DE KEGHEL. *Rev. chim. ind.* **29**, 285-8(1920).—Dry about 10 g. of the finely powdered material at 105° to const. wt. Transfer 2 g. of the dried material to a suitable flask and insert a stopper carrying a separatory funnel and an outlet tube connected with a condenser and a dephlegmating column. Add (through funnel) 7 cc. of SnCl_2 soln. (40 g. $\text{SnCl}_2 + 750$ cc. concd. $\text{HCl} + 250$ cc. H_2O), rinse the funnel with 20 cc. HCl (1:1) and run this into the flask. Pass a current of CO_2 through the app. for 5 min., heat just below the b. p. for 20 min., maintaining the CO_2 current. Absorb the vapors in 3 100 cc. flasks connected in series and containing in order (1) 50 cc. $\text{H}_2\text{O} + 2$ cc. glacial AcOH ; (2) 30 cc. $(\text{AcO})_2\text{Cd}$ 25 g. $(\text{AcO})_2\text{Cd} + 200$ cc. glacial $\text{AcOH} + 800$ cc. H_2O + 20 cc. H_2O ;

(3) 20 cc. $(\text{AcO})_2\text{Cd}$ + 30 cc. H_2O . Boil gently for exactly 10 min. and let cool in the current of CO_2 ; unite the contents of the absorption flasks in an 800 cc. beaker, expel the CO_2 , rinse the tubes and flasks with cold freshly hoiled H_2O containing 1% AcOH to remove all CdS . Add 20 cc. 0.1 N I soln., stir, cover and let stand exactly 12 min.; dil. freely with H_2O and titrate the excess of I with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ soln. and starch.

F. W. SMITHER

A new method for the estimation of carbon dioxide in alkali bicarbonate in the presence of carbonate. W. HARTMANN. Erlangen. *Z. anal. Chem.* 59, 289-97 (1920); cf. *C. A.* 14, 2957 for the method.—No less than 65 results are given in full detail, showing the wide applicability of the method.

ALBERT SALATHE

The removal of nitrates by means of alcohol. R. SCHNEIDEWIND. *Chem. Met. Eng.* 24, 22 (1921).—Instead of evap. with H_2SO_4 to fumes, the HNO_3 may be removed by reduction with alc. To the hot soln. containing 20 cc. concd. HNO_3 in 150 cc. of liquid add 15 cc. of concd. H_2SO_4 and 5 cc. of alc. and heat carefully. From time to time add further portions of alc. until finally no more red fumes are obtained. During this treatment, cuprous salt is sometimes pptd. when Cu is present.

W. T. H.

The determination of nitrogen in nitrites and nitrates by means of copper-magnesium. TH. ARND. *Z. angew. Chem.* 33, I, 296-8 (1920); cf. *C. A.* 12, 662.—Tests were made to det. the limitations of the method which depends on the distillation of the neutral salt with Cu-Mg alloy in the presence of MgCl_2 and catching the ammoniacal distillate in a measured vol. of standard acid. Some commercial fertilizers contain NaCl , KCl and CaSO_4 . The presence of these salts is shown to have no effect on the results. On the other hand, if considerable Na_2SO_4 is present the results are low. This error was overcome by increasing the MgCl_2 dosage from 5-50 cc. As was to be expected, N present as NH_4 salt was obtained by the method. In the analysis of ammonium sulfate-nitrate mixtures, however, it is advisable to use 0.5 g. of the sample for analysis, not less than 5 g. of Cu-Mg alloy and 50 cc. of 20% MgCl_2 soln. In carrying out this method, it is important to remember that the presence of free alkali nullifies the reducing power of the alloy.

W. T. H.

The determination of cyanamide nitrogen in commercial calcium cyanamide. H. NEUBAUER. Agricultural Expt. Station, Bonn. *Z. angew. Chem.* 33, I, 247-8, 254-6 (1920).—Calcium cyanamide deteriorates on standing with the formation of dicyanamidine which is harmful rather than good for plants. The purpose of this work was to establish an improvement in the method of estimating the available nitrogen of commercial fertilizer containing Ca cyanamide. 1. *Detn. of cyanamide N by the H_2 method:* Extract with 100 cc. of acetone to remove dicyanamide, urea and free cyanamide, using 20 cc. portions and allowing about 20 min. for each treatment. Treat the residue in a flask with 3 g. of Cu-Mg alloy (60% Cu), 5 cc. of 20% MgCl_2 soln. and 15 cc. glacial AcOH , taking precautions to prevent loss of soln. by using a tube as reflux condenser. After 10 min. rinse down the condenser and sides of the flask with water, add 3 g. more of the alloy and allow the reduction to proceed for another 10 min. Rinse as before, dil. to 300 cc., add a little paraffin to prevent too much foaming, a slight excess of caustic soda and distill off the NH_3 formed from the calcium cyanamide into a known vol. of standard acid. If the original sample is badly deteriorated, the N value now obtained will be a little too high. A correction factor may be applied by detg. the total N in the acetone-insol. part of another portion of material and multiplying the difference between the 2 N values by 0.25 and subtracting this product from the value obtained by the above procedure. 2. *Testing and recovery of acetone:* The acetone must be perfectly dry as shown by CuSO_4 test, because if water is present some of the Ca cyanamide will be dissolved. Shake the used acetone with oxalic acid to neutralize NH_3 and distill, keeping the temp. down to 58° . Dry over K_2CO_3 and again distill at not over 58° . 3. *Detn. of N in acetone ext:* If it is desired to det. N in the ace-

tone ext., place the acetone ext. in a Kjeldahl flask, add 0.5 g. of oxalic acid to combine with NH_3 , and after a short time distill off the acetone and det. N by the Kjeldahl method. 4. *Detn. of N adsorbed by C*: Apparently the presence of free C in the sample causes retention of some of the available N. To det. this, take the residue from the NH_3 distillation in (1) and wash it several times with dil. HCl. Then det. the N in the washed residue by the Kjeldahl method. The N values thus obtained range from 0.08 to 0.16%.

W. T. H.

The determination of nitrites and nitrates in plant tissue. W. H. STROWD. *Soil Sci.* 10, 333-42(1920).—The Caron method (*C. A.* 6, 2246) and the nitron method of Busch are unsatisfactory for the detn. of NO_2 in plant tissue. The Kjeldahl-Gunning-Arnold method and Kjeldahl method modified to include NO_2 are unsatisfactory. The Devarda and Schloesing methods with modifications can be used to det. NO_2 in plant tissue. A satisfactory procedure to det. NO_2 and NO_3 in plant tissue is as follows: Dilute 2 aliquots of a cold-water ext. of plant tissue to 250 cc. in a Kjeldahl flask. Add a piece of paraffin and 2.5 g. of NaOH in concd. soln. To 1 soln. add 1 g. of Devarda's alloy. Attach to distg. app. and heat over low flame 1 hr. or until action stops and distill exactly 150 cc. Titrate with 0.0357 *N* alkali. The difference gives N as NO_2^- and NO_3^- . Make a similar portion of the cold-water ext. up to 25 cc. and add 0.15 g. of aspartic acid or more. Heat on water bath for 1 hr. Divide into 2 equal portions and treat as above. The difference between the first and 2nd results represents the NO_2^- .

W. J. ROBBINS

Addition to the method for determining mercury in organic substances. MAURICE FRANÇOIS. *Bull. soc. chim.* 27, 568(1920); cf. *C. A.* 14, 2144.—In detg. Hg in org. compds. by pptn. with Zn, the presence of alc., ether and HCl is advantageous. The new directions read as follows: Weigh a 125 cc. Erlenmeyer, introduce about 0.5 g. of dry powdered substance and again weigh. Add successively 30 cc. ether, 10 cc. alc. and 1 cc. concd. HCl. Introduce 1 g. of pure Zn filings, agitate and allow to stand 30 min. Repeat the treatment with Zn twice more. Allow to stand 24 hrs. in a cool place and then proceed as in the former directions.

W. T. H.

The use of reduced copper in the elementary analysis of organic substances. ÉMILE CHERBULIEZ. *Helvetica Chim. Acta* 3, 652-3(1920).—C. confirms the conclusion of Perrot (cf. *Compt. rend.* 48, 53(1859)) that Cu containing appreciable amts. of Fe or Zn should not be used in detg. N. At a red heat such alloys reduce CO_2 to CO, while alloys of Cu with notable amts. of Sn showed no ill effect.

F. W. SMITHER

A sensitive form of the Lieben iodoform reaction. RUDOLF KUNZ. *Z. anal. Chem.* 59, 302-3(1920); cf. *Z. anal. Chem.* 9, 265(1870).—To 10 cc. of the liquid to be tested, add 1.5-2 cc. 10% NaOH soln., about 0.15 g. KI and 0.2 g. $\text{K}_2\text{S}_2\text{O}_8$ and heat to 50-60°. Should the 10 cc. of soln. used contain 1 drop of alc., a ppt. of CHI_3 forms after heating 1 min. and increases on further heating for a short time. With 10 cc. of a soln. containing 1 drop of alc. in 50 cc. H_2O , a ppt. forms after 5 min., with 10 cc. of 100 cc. H_2O + 1 drop of alc., after 10 min.

F. W. SMITHER

The determination of hydrocyanic acid. R. LEITCH MORRIS. *Am. J. Pharm.* 92, 908-16(1920).—See *C. A.* 14, 2897.

W. G. GAESSLER

The determination of acetic acid in pyroigneous acid. V. E. GROTLISCH. *J. Ind. Eng. Chem.* 12, 1183-6(1920).—G. describes a procedure based on distn. with xylene, elimination of tars, phenols, etc., in the distillate by oxidation with alk. KMnO_4 , and then redistn. with H_3PO_4 .

F. W. SMITHER

Determination of aniline vapors in the air. MIRIAM S. ISZARD. *J. Ind. Hyg.* 2, 259-66(1920).—The samples were collected by emptying carboys filled with water and allowing air to enter, whereupon the aniline was absorbed with 200 cc. of 10% HCl or a measured vol. of air was passed through two Pettenkofer tubes filled with 10% HCl and the resulting aniline-HCl titrated slowly with 1% NaNO_2 at 5-8°, until a

drop gave an immediate blue color with KI-starch paper. The soln. was then warmed to 12° and the titration continued slowly until it again gave an immediate blue color with KI-starch paper. The factor used is derived from checking against a standard of pure aniline.

H. V. ATKINSON

Preliminary note on the action of fluorine on potassium bisulfate (BRUNNER) 6. Calibration of sixteen Lovibond red glasses (PRIEST) r.

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8—MINERALOGICAL AND GEOLOGICAL CHEMISTRY

EDGAR T. WHERRY AND WALTER F. HUNT

Is the manner of attachment of a crystal to its support determined by some law, or is it accidental? GEORG KALB. *Centr. Min. Geol.* 1920, 65-7.—K. presents a tabulation of the mode of attachment of a series of minerals to their support, data taken from current text-books on mineralogy. The fact is brought out that needle-shaped, bladed and prismatic crystals are attached with their longer dimension normal to their support, while platy crystals are attached by an edge. This is true even if the support

is non-cryst. K. concludes: (1) that "crystals have a tendency to attach themselves to their supports so that a predominating rational direction is normal to the support." NaCl crystallizes from an aqueous soln. in cubes which are attached to the sides and bottom of the vessel by their cube faces. Crystg., however, from a NaOH solution the cubes are so attached that a trigonal axis stands normal to the support. K. therefore concludes (2) that "the plane of attachment is a crystal face having a high surface tension." Referring further to the observation of Becke that in the case of penetration twins the mode of attachment is such that the line of symmetry between the two individuals stands normal to the support, K. finally concludes (3) that "crystals that are permitted to grow undisturbed assume a position of equilibrium with respect to their support, which is detd. by their surface tension." OTTO VON SCHLICHTEN

Plazolite, a new mineral. WILLIAM F. FOSHAG. U. S. National Museum. *Am. Mineral.* 5, 183-5(1920).—Small colorless dodecahedrons on vesuvianite from Riverside, California, prove to be a new species, and are named from the Greek *plazo*, to perplex, because of the difficulty in interpreting the compn. The mineral was formed at a late stage of contact metamorphism, by the action of hot magmatic waters on earlier formed minerals. Its $d. = 3.129$, $H. = 6.5$, and n by immersion method $= 1.710$. Considerable variation was shown by the 3 analyses, even though all were made on carefully selected transparent cryst. material: SiO_2 23.85-25.06, Al_2O_3 22.77-24.63, CaO 40.13-40.22, MgO none to 0.12, H_2O 9.04-9.39, and CO_2 1.13-3.41%. The CO_2 and SiO_2 seem to vary reciprocally, and if calcd. together the ratios become $SiO_2:Al_2O_3:CaO:H_2O = 1.8:1.0:2.9:2.1$, giving roughly the formula $3CaO.Al_2O_3 \cdot 2(SiO_2.CO_2) \cdot 2H_2O$. The mineral is thus highly basic, but appears to be related to sodalite.

E. T. W.

Armangite, a new arsenite from Långbanshyttan. G. AMINOFF AND R. MAUZELIUS. *Geol. För. Förh.* 42, 301-9(1920).—A new arsenite of Mn was found by G. Flink at Långbanshyttan, Sweden, in 1919. The name is derived from its constituents. It occurs in a cryst. mixture of calcite and barite accompanied by small amts. of pale green fluorite, small black rhombohedra of hematite, a brown, optically uniaxial, negative, lamellar mineral, and a S-yellow orthorhombic (?) mineral resembling ecdemite. Armangite is black with brown streak, brown to yellow in thin section and generally as rhombohedral crystals, with more or less pronounced prismatic habit. Two tables give measurements made on several crystals. $H=4$. Optically uniaxial.—The $n's$ are higher than a soln. of S in methylene iodide ($n = 1.79$), lower than amorphous S ($n = 1.93$), while the birefringence is weak. Sp. gr. $= 4.43$. Analysis of 3 portions (0.5-0.8 g.) gave: As_2O_3 42.92, Sb_2O_3 0.40, PbO 0.32, FeO 2.19, MnO 45.06, CaO 2.83, MgO 0.49, H_2O 0.71, CO_2 5.08, insol. 0.20, sum 100.20%. It is sol. without difficulty in HCl, sepg. As_2O_3 . Detn. of H_2O changed the mineral to a grayish-black porous fused glass. An attempt was made to det. the degree of oxidation of the As, but with unsatisfactory results. The Fe was assumed to be ferrous, though some of it is probably ferric, the HCl soln. of the mineral being yellow. Material for analysis had been carefully selected, and calcite sepd. by suspension in methylene iodide (sp. gr. 3.33); but the heavier material still contained calcite-like material which could not be further sepd. To det. whether the 5% of CO_2 belonged to the mineral or to the impurity, some tens of grains about 0.5 mm. in diam. were put into HCl one after another. Some grains evolved abundant CO_2 , others CO_2 from some corners and edges, while the bulk of them dissolved without evolving much CO_2 . The amt. of CO_2 is greater than corresponds to the amts. of CaO and MgO, so the mineral must contain some carbonate of Mn (and Fe?). The results of the analysis indicate the mineral to be an ortho-arsenite of Mn, $Mn_3(AsO_3)_2$, with a small part of the Mn substituted by H; and the mixture of carbonates in the material to be composed of: MnO 3.69, CaO 2.83, MgO 0.49, CO_2 5.08, total 12.09.

W. SEGERBLÖM

Unusual minerals in limestone near York, Pa. MORTON L. JANDORE. *Am. Mineral.* 5, 196(1920).—Sphalerite occurs in a stratum of apparently unmetamorphosed limestone, together with a series of alteration products provisionally identified as greenockite (xanthochroite), smithsonite, aurichalcite and hydrozincite. E. T. W.

Minerals in the Niagara limestone of western New York. ALBERT W. GILES. Univ. Va. *Proc. Rochester Acad. Sci.* 6, 57-72(1920).—The properties, modes of occurrence, etc., are given for 20 minerals which occur in this formation. They are chiefly carbonates, sulfates, and sulfides, with 2 oxides and one element, S. Brief notes on their origin are appended. E. T. W.

Notes on anglesite, anthophyllite, calcite, datolite, sillimanite, stilpnomelane, tetrahedrite and triplite. EARL V. SHANNON. *Proc. U. S. Nat. Museum* 58, 437-63 (1920).—Three lots of anglesite are described crystallographically. A somewhat unique calcite from Idaho is described. Tetrahedrite from Hypotheek mine, Idaho, gave on analysis: SiO_2 13.57, Sh 23.03, Fe 4.41, Cu 32.38, Zn 3.33, S 22.74, sum 99.46%. It is remarkable that but traces of Ag and As are present, since As is in arsenopyrite in the same vein. Triplite from Chatham, Conn., is described and compared with triplite from Nevada both as to chem. compn. and optical properties. The Conn. samples showed higher ns . and a partial replacement of Mn by Fe. Datolite from Meriden, Conn., is given a crystallographic description. Gedrite, a variety of anthophyllite, from Chesterfield, Mass., gave on analysis: SiO_2 49.66, Al_2O_3 6.74, Fe_2O_3 1.23, FeO 18.09, CaO 3.38, MgO 18.62, MnO 0.05, H_2O 1.58, sum 99.33%. A hucholzite variety of sillimanite in pegmatite from Russell, Mass., contained: SiO_2 38.10, Al_2O_3 59.48, Fe_2O_3 1.05, ignition 1.32, sum 99.95%. Stilpnomelane from Rocky Hill, N. J., contained: SiO_2 45.12, Al_2O_3 6.29, Fe_2O_3 23.67, FeO 5.93, MgO 9.36, H_2O 9.12%. A sample from Westfield, Mass., gave SiO_2 44.08, Al_2O_3 4.74, Fe_2O_3 5.27, FeO 23.31, MgO 8.36, MnO 0.87, H_2O 12.49%. These 2 samples vary mainly in the degree of oxidation of their Fe. L. W. RIGGS

Commercial minerals of California. W. O. CASTELLO. California State Mining Bureau, *Bull.* 87, 124 pp.—The distribution, compn., tests, metallurgy and uses of the minerals of California are given. ALBERT R. MERZ

Preliminary report on the deposits of manganese ore in the Batesville District, Arkansas. HUGH D. MISER. U. S. Geol. Survey, *Bull.* 715G, 93-124(1920); cf. *C. A.* 14, 715.—These deposits have been worked at times since 1849 but the periods of chief production of high grade ores were 1885-1898 and 1915-1918. Since 1904 there has been quite a steady production of ferruginous Mn ores containing 10 to 30% of Mn. The ores are found as 6 different oxides, the Mn content ranging from 70% in hausmannite to 20-30 in wad. The deposits are described as replacement, residual and transported ores. The av. compn. of more than 350 carloads shipped in 1917-1918 was about Mn 47.0, Fe 5.7, P 0.30, SiO_2 7.0, Al_2O_3 2.98%. L. W. RIGGS

Marble resources of southeastern Alaska. ERNEST F. BURCHARD. U. S. Geol. Survey, *Bull.* 682, 14 pp.(1920).—The geographical and geological features are described by Theodore Chapin. The origin and chem. characters of the marble are quite fully discussed and more than 130 samples, classified according to color, are tabulated. Alaskan marbles are noted for their variety of color and are used for interior finish and decoration. Over 70 buildings in the U. S. are listed as containing Alaskan marble. L. W. RIGGS

Report on road materials along the St. Lawrence River, from the Quebec boundary line to Cardinal, Ontario. R. H. PITCHER. Canada Dept. Mines, Mines Branch, *Bull.* 32, 65 pp.(1920).—The Survey was undertaken in order to secure information regarding available road materials for the proposed improved highway between Montreal and Toronto. The rock outcrops consist of dolomites and limestones. Large deposits

of glacial material occur. The characters of these materials are described, with the results of lab. tests.

S. G. GORDON

Natural gas resources available to Dallas and other cities of central north Texas. E. W. SHAW AND P. L. PORTS. U. S. Geol. Survey, *Bull.* 716D, 55-89(1920).—The territory considered is located within a radius of 200 miles from Dallas. Owing to shortage of gas during 1919 a special recent study of this field was made during the winter of 1919-20, and the estd. recoverable reserves of gas on Jan. 1, 1920, were placed at 200 billion cu. ft. By rigid economy the supply may last 6 to 10 years, but with shortages nearly every winter.

L. W. RIGGS

Rhyolites of Lipari. HENRY S. WASHINGTON. *Am. J. Sci.* 50, 446-62(1920).—Rhyolite obsidian was jet black, with vitreous luster and generally perfect conchoidal fracture. The sp. gr. of sample from Forgia Vecchia = 2.363, that from Rocche Rosse 2.370. The n_s = 1.490 and 1.488-9, resp. Analyses of these specimens and a sample of pumice from Mt. Pelato agreed quite closely except that the pumice gave 2-3% less SiO_2 and 2-3% more H_2O . The figures for the obsidian from Forgia Vecchia were: SiO_2 73.86, Al_2O_3 12.49, Fe_2O_3 0.60, FeO 1.40, MgO 0.11, CaO 0.75, Na_2O 4.05, K_2O 4.81, H_2O +0.92, H_2O -0.09, TiO_2 0.12, ZrO_2 0.06, SO_2 0.06, Cl 0.26, sum 99.58%. On calcn. of the norms this specimen has the symbol I.'4.1.3.; the Rocche Rosse obsidian and the Mt. Pelato pumice each I.4.1.3. A sample of lithoidal rhyolite from Mt. Giardina gave I'.4.1'.3 and one from Mt. Guardia I(II).4.1(2).3.; the latter being 5.06% lower in SiO_2 but higher in the oxides of Fe, Mg and Ca. A specimen of hyalodacite from La Perrera showed 13% less SiO_2 than the Forgia Vecchia obsidian which was more than made up by increased Al_2O_3 and CaO . Symbol: I.4(5).-3.3. Rhyolite obsidian from the Island of Milos gave higher SiO_2 and CaO . Symbol: 1.3(4).2.3(4). From W.'s analyses of Italian lavas and those of others (U. S. Geol. Survey, *Prof. Paper* No. 99) it appears "that there is a direct relation between the ratios of the Fe oxides and the degree of crystallinity in the rhyolites and basalts of Italian volcanoes, in the sense that FeO dominates over Fe_2O_3 in the glassy forms, while the reverse is true in the cryst. forms of lavas that are otherwise of almost identical compn. and are derived from the same magma." Various suggestions are made with reference to possible reactions between Fe compds. and the magmatic gases.

L. W. RIGGS

The basalts of the "Blaue Kuppe bei Eschwege" and adjacent occurrences, and their cristobalite. PAUL RAMDOHR. *Centr. Min. Geol.* 1920, 33-6.—The basalt is fine-grained and composed of olivine, augite, plagioclase, feldspar, ores, apatite, a little biotite and hornblende. At the contact with the Buntsandstein the blocks of the latter that have become engulfed in the basalt have been profoundly altered, with the formation of new minerals, such as cordierite, pyroxenes, ores and feldspars. Pneumatolytic minerals are magnetite, specular hematite, cristobalite, tridymite, chalcedony, apatite, hypersthene, augite, feldspar, mica and titanite. R. describes the cristobalite in detail. Crystals are generally smaller than 1 mm. and milky white. They occur in 3 habits: (1) Well shaped octahedra; (2) 6-sided plates, and (3) crystals that are geometrically identical with tridymite. All become isotropic at 235-250° C.; d_s = 2.290-2.320, n = 1.485; birefringence = 0.0018. There are all transitions between habit 1 and 2, the latter being the commoner. The transitions, as well as twinning according to the spinel law, indicate that habit 2 represents distorted octahedra. There are also transitions between habits 2 and 3 and closer investigation of the apparent hexagonal prisms reveal that they are built up by an oscillatory combination of the octahedron and cube. Interpenetration twins of the tridymite type after (1016) and (3084) also occur and are shown to be due to distortion of different octahedral faces of 2 octahedrons in parallel position.

OTTO VON SCHLICHTEN

Radium content of the rocks of the Loetschberg tunnel (POOLE) 3. Apparatus for grinding rock specimens (HADDING) 1.

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9—METALLURGY AND METALLOGRAPHY

WILLIAM BRADY, ROBERT S. WILLIAMS

A résumé of literature on the theory of flotation with critical notes. H. R. ADAM. *J. Chem. Met. Mining Soc. S. Africa* 21, 62 (1920); *Mining Sci. Press* 121, 765-70 (1920).—An addition to the paper referred to in *C. A.* 14, 3625. E. J. C.

Résumé of literature on the theory of flotation. H. R. ADAM. *Mining Sci. Press* 121, 705-70 (1920).—See *C. A.* 14, 3625. E. H.

The Greenawalt sintering process. WILLIAM E. GREENAWALT. *Mining Sci. Press* 122, 81-5 (1921).—The Greenawalt process is intermittent in operation, a charge being subjected to a down-draft blast of air, and after ignition the sintering action proceeds from the top downward. It is simple and present plants range from 20 to 1000 tons per day capacity. The charge to be sintered should have about 10% moisture, and should be properly mixed. The S content of Cu and Pb ores may vary from 5 to 15%, and the coke content of Fe ores should be between 3 and 10%. A 10 by 24 ft. pan, the largest unit, has a capacity of 150 to 250 tons daily. The charge car spreads a layer of non-sinterable material over the grates to a depth of $\frac{1}{4}$ to $\frac{3}{4}$ in. before dumping the charge, all of which is done in a few secs. The ignition requires about a min. and either gas or oil may be used; with the latter about 1 gal. is used per ton of sinter. The time of sintering varies from 15 to 60 min., and the suction used from 10 to 35 in. of water. About 5 to 6.5 kw. hr. is used per ton of sinter. Sintering may replace drying of flotation concentrates, first, because the moisture contained is necessary for sintering; second, the concentrates furnish their own fuel for sintering, as against purchased fuel required for drying; third, reduction in wt. for shipping and smelting, of from 15 to 20%; fourth, the saving in sacking where the fine concentrates are sacked to prevent loss. The combination of sintering and blast furnace smelting bids fair to compete with roasting and reverberatory smelting. A. H. HELLER

The Bunker Hill smelter. T. A. RICKARD. *Mining Sci. Press* 122, 43-60 (1921).—The plant consists of crushing and sampling mill, Dwight Lloyd sintering machines, Traylor blast furnaces, Pb and Ag refineries. The sintering plant uses pulverized coal as fuel. The fume and gases from the sintering plant pass to Cottrell precipitators. The roast, flux, ore and coke are all drawn from sep. bins and weighed before being

charged to the blast furnaces, of which there are 4, having a combined capacity of 1200 tons per day. The charge contains 32% Pb and less than 4% Zn. The slag and mat are drawn off intermittently into pots, while the lead is run off continuously. Fume from the blast furnaces passes through settling chambers to a bag house. The product from the bag house contains 72% Pb and 8% S. The temp. of drossing is about 360° and in softening where the Sb is removed a temp. of 920° is maintained for 20 hrs. Zn is used for desilverizing, and the resulting precious metal alloy skimmings are run through a Howard press for removal of Pb. The refined Pb is cast into pigs by a Miller casting machine. The pressed alloy is retorted for removal of the Zn and the final retort metal is sent to the Ag refinery, where through the use of a Rhodes cupelling furnace any remaining Pb is driven off. The Ag ingots are parted in iron kettles with 66° H₂SO₄ and the Ag in the resulting sulfate is deposited upon Cu. The final CuSO₄ is evapd. to crystals. The Pb refinery is equipped with a reverberatory furnace for the treatment of various drosses. A description is given of the coal pulverizing plant, which uses the Bonnet system.

A. H. HELLER

Crystalline slag obtained from the basic Martin process. OTOKAR QUADRAT. *Chem. Listy* 14, 73-6(1920).—The crystals were formed under the following conditions. A slag, containing about 66% Fe, was discharged into a cooling pit. This iron cooling slowly kept the supernatant layers of slag in a molten condition for a considerable period. The slag cooling slowly allowed crystn. to occur. Mineral. examn. of the crystals by B. Ježek and M. Ulrich showed that they belonged to the rhombic system. The following measurements of angles and faces were obtained: $a:m = 41^\circ 1'$, $m:m' = 82^\circ 2'$, $d:d' = 47^\circ 20'$, where $m = (021)$, $2P\infty$; $a = (010)$, $\infty P\infty$; $d = \infty P$, (110) . The sp. gr. equaled 3.04. These results do not identify the crystals with any known silicates. Many of the crystals were twinned. The size of single crystals varied from 5 mm. to 0.5-0.6 mm. Some were colorless to yellowish, transparent and of very high luster. The results of analysis indicated that their compn. would be best expressed by the following formulas: $(\text{FeOMnO})_2\text{SiO}_4 \cdot 4(\text{CaOMgO})_2\text{SiO}_3$ or $(\text{FeOMnOMgO})_2\text{SiO}_3 \cdot 2(\text{CaO})_2\text{SiO}_3$. The analysis of the original slag gave: SiO₂ 19.78, FeO 14.35, Fe₂O₃ 3.10, MnO 16.05, Al₂O₃ 2.52, CaO 35.85, P₂O₅ 3.99, MgO (by difference) 4.04, S 0.32%.

JOHN M. KRNO

The platinum metals. A. D. LUMB. (Brit.) Imperial Inst., Mineral Resources Comm., *Monograph; Chem. Trade J.* 67, 799-801(1920).—A comprehensive survey of the subject covering occurrences, properties, metallurgy, uses, alloys, particularly alloy substitutes for Pt, and production.

E. J. C.

Felix Robin. LÉON GUILLET. *Rev. métal.* 17, 696-7(1920).—A biographical note.

E. H.

Tests on a new Krupp soft iron. P. GOERENS AND FR. P. FISCHER. *Elektrochem. Z.* 27, 1-11(1920).—A detailed account is given of tests made on a practically pure Fe produced in the Siemens-Martin furnace. The properties of this "WW" brand Fe approach those of Cu. The elastic limit is 20-23 kg/mm², as compared with 4 kg/mm² for Cu; tensile strength 31 kg/mm², as compared with 21.5 kg/mm². Analysis of the iron gave C 0.057, Si 0.01, Mn 0.097, P 0.01, S 0.021%.

C. G. F.

An electrometric method for detecting segregation of dissolved impurities in steel. E. G. MAHIN AND R. E. BREWER. *J. Ind. Eng. Chem.* 12, 1095-8(1920).—A method has been developed for measuring the electrode potential of a single grain or microscopic point on a metal specimen. Results so far obtained show lower values than are given when a pure metal is immersed according to the usual procedure. When the method is applied to a piece of steel containing a segregated ring of ferrite, produced by heating in contact with Al bronze, it is found that av. values for the ferrite ring are 0.051 v. lower than for ferrite in the unaffected body of the material. This indicates a differ-

ent degree of purity for ferrite under the 2 conditions. An attempt will be made to improve the method.

A. L. FIELD

Inclusions and ferrite crystallization in steel. II. Solubility of inclusions. E. G. MAHIN AND E. H. HARTWIG. *J. Ind. Eng. Chem.* 12, 1090-5(1920); cf. *C. A.* 13, 2181.—From expts. described, the hypothesis is advanced that the special element, elements, or compds. have diffused into the surrounding steel, and that they have there exerted an influence toward throwing ferrite out of the austenite soln. when the latter cools into the transformation range. The expts. possibly throw some light upon the character of the influence of non-metallic inclusions upon ferrite segregation. The effect of P upon C distribution is then but one illustration of the general law. Practically all of the elements that can enter steel exert a similar influence. While the inclusion furnishes a continuous supply of the dissolved impurity and no amt. of heat treatment can cure its evil effects, the opposite is true with the dissolved element or compd. which is not associated with discrete particles of inclusions.

A. L. FIELD

Influence of antimony and arsenic on admiralty gun metal. R. T. ROLFE. *Engineering* 110, 689-91, 752-4(1920).—See *C. A.* 14, 3215.

E. H.

Constitution of the alloys of aluminium and magnesium. D. HANSON AND M. L. V. GAYLER. *Engineering* 110, 788-91, 819-21(1920).—See *C. A.* 14, 3212.

E. H.

Studies on the corrosive action of chlorine-treated water. I. The effects of steel on the equilibrium: $\text{Cl}_2 + \text{H}_2\text{O} + \text{HCl} + \text{HClO}$ and of products of the equilibrium on steel. GEORGE L. CLARK AND R. B. ISELEY. *J. Ind. Eng. Chem.* 12, 1116-22 (1920).—In the solns. containing no steel final equil. is reached after 48 hrs., the equil. const. for the reaction given in the title being 0.467 at 25° when the equil. concn. of Cl_2 is 0.00006 mol. per l. In the solns. in contact with steel the HClO disappears some time after 24 hrs. and after 72 hrs. the Cl_2 disappears, forming HCl , which latter acid remains at const. concn. thereafter. This indicates that finally only one equil. is involved, viz.: $\text{FeCl}_2 + 3\text{H}_2\text{O} \rightleftharpoons \text{Fe}(\text{OH})_3 + 3\text{HCl}$. From detns. on the wt. loss of low-C steel due to corrosion over a period of 100 days, in which city water, river water, and distd. water, with and without added Cl , were used, it is concluded that the dissolved electrolytes exert a profound influence upon the Cl equil. and upon the Fe oxidations. This paper is preliminary to more practical work on the corrosive action of Cl -treated H_2O upon living organisms and upon metal containers and pipes.

A. L. FIELD

Electric welding. C. J. JEWELL. *Electrician* 85, 680-1(1921).—A review.

C. G. F.

Some aspects of electrolysis (corrosion) (ALLEMAN) 4.

DICHMANN, CARL: *Der basische Herdofenprozess*. 2nd Ed. revized. Berlin: Julius Springer. 278 pp. M. 42, bound M. 50. For review see *Z. angew. Chem.* 33, II, 472(1920).

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FOSTER, FRANK: *Foster's Pocket Reference Book on Iron and Steel Works*. London: Iliffe & Sons. 130 pp. 2s. For review see *Tech. Review* 6, 101(1920).

FRANKE, FRANZ WERNER: *Abriss der neuesten Wirtschaftsgeschichte des Kupfers*. Leipzig: Duncker & Humblot. M. 32. For review see *Z. angew. Chem.* 33, II, 440(1920).

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GOODALE, S. L.: *Chronology of Iron and Steel*. Pittsburgh, Pa.: J. Ramsey Speer. 300 pp. For review see *Manufactures Record* 79, No. 2, 132(1921).

GRAHN: *Die wichtigsten Aufbereitungsverfahren unter besonderer Berücksichtigung der Steinkohlen und der Erze*. Leipzig: Max Jänecke. 79 pp. M. 7.95.

HULLE, FR. W.: *Die Grundzüge der Werkzeugmaschinen und der Metallbearbeitung*. 2nd Ed. enlarged. Berlin: Julius Springer. 210 pp. M. 11. For review see *Z. ver. deut. Ing.* 64, 922(1920).

OSANN, BERNHARD: *Lehrbuch der Eisen- und Stahlgiesserei*. 4th Ed. Leipzig: Wilh. Engelmann. 672 pp. M. 63, bound M. 81.

PAGÉ, VICTOR W.: *Modern Welding Methods*. New York: Norman W. Henley Publishing Co. 292 pp. For review see *Iron Age* 106, 1721(1920).

SCHOTT, ERNST A.: *Die Metallgiesserei*. 2nd Ed. revized and enlarged. Leipzig: Bernb. F. Voigt. 397 pp. M. 37.50. For review see *Stahl u. Eisen* 40, 1469(1920).

Ore-flotation apparatus. J. T. TERRY, JR. U. S. 1,362,370, Dec. 14.

Concentrating ores. W. M. MARTIN. Brit. 150,113, June 16, 1919. In concg. app. of the kind in which pulp is passed over frosted or fluted or frost-fluted glass surfaces, the pulp is caused to flow intermittingly, and H_2O may be supplied to the surface between successive flows of pulp. The grass may be arranged in steps to cause the pulp to cascade during its flow. A suitable construction is specified.

Smelting. J. LUND. U. S. 1,360,552, Nov. 30. In smelting ores or metals such as Fe, a puffed slag is introduced into the charge, the pores of the slag being used as carriers for a filling of Na_2CO_3 and petroleum coke, serving as flux and reducing agent and preventing too great decarbazionization.

Smelting ores. E. H. HAMILTON. U. S. 1,362,570, Dec. 14. In smelting ores such as Ph ores a blast of air and finely divided fuel is introduced into the charge at a point within the interior wall of the furnace so as to prevent escape of the fuel up the walls of the furnace.

Extracting copper from ores. L. D. MILLS. U. S. 1,360,666, Nov. 30. Finely divided freshly reduced Fe is used as a means for pptg. Cu from solns. such as are obtained by the treatment of a Cu ore with H_2SO_4 or $NaHSO_4$. The use of Fe in this form has the advantage that Fe from reduced Fe ore can be used (still carrying gang which is afterwards sepd. by flotation).

Extracting bismuth from ores. J. F. CULLEN and M. J. UDY. U. S. 1,360,271, Nov. 30. Crusbed calcined Bi ore such as carbonate or oxidized sulfide is leached with brine containing an acid such as H_2SO_4 to obtain a soln. of a Bi salt which may be filtered and passed into a pptg. tower filled with Fe scrap to ppt. Bi as a metallic sponge.

Obtaining zinc from ores. F. LAIST and J. O. ELYON. U. S. 1,362,166, Dec. 14. Zn ore is leached with dil. H_2SO_4 in insufficient quantity completely to dissolve the Zn content of the ore. The resulting $ZnSO_4$ soln. is purified and electrolyzed with insol. anodes to produce Zn and a dil. acid soln. A portion of the acid thus obtained is used for leaching additional ore and another portion for leaching residue from the first leaching operation.

Extracting gold, silver and copper from ores. G. T. HANSEN. U. S. 1,361,459, Dec. 7. Au, Ag and Cu are recovered from their ores by leaching the ore with KCN soln. at ordinary temp., heating the soln. to about 32° or higher and subjecting the hot soln. to the action of Zn or Al to ppt. the Cu. The Au and Ag are recovered by pptn. with the Cu or by a previous pptn. from the cold soln.

Extracting metals. A. E. BOURCOUD. Brit. 151,644, Sept. 27, 1920. See U. S. 1,344,977 (C. A. 14, 2467).

Recovering tin and zinc from scrap. G. H. CLEGG. Brit. 151,374, June 25, 1919. ZnCl_2 , palmitin, and Sn ore are recovered from the scruff from tinning pots. The scruff is crushed to pieces about $\frac{1}{4}$ in. in diam., agitated in a tank with about $\frac{1}{4}$ as much H_2O below 60°F . until the ZnCl_2 soln obtained has a sp. gr. of 1.3, when it is drawn off and filtered in a filter press. The remaining sludge in the tank contains "palmitine," which term includes palmitin and palmitic acid, Sn, SnO and a small proportion of ZnCl_2 and is ground with H_2O to a paste and screened to remove the heavier particles of Sn. "Sn soap," which consists of SnO and "palmitine," and a proportion of the lighter particles of Sn are also sepd. in the screening operation and are then agitated in a vessel when the Sn sinks and the supernatant "palmitine" and SnO are filtered to remove H_2O , treated with sufficient HCl to dissolve any Zn and Sn oxychlorides, filtered, dried and distd. The distillate contains a mixt. of palmitine and oil.

Furnace for reducing ores and similar purposes. R. J. ANNERSON. U. S. 1,362,212, Dec. 14. The furnace is adapted for smelting Cu, Pb or Fe ores or other materials.

Reversible open-hearth furnace. C. D. FULLER. U. S. 1,362,412, Dec. 14.

Pulverizing metals. J. P. MELLOR. Brit. 150,400, July 4, 1919. Zn or other metal is reduced to powder by heating the metal to the highest possible temp. it can attain without volatilization, and pouring it in a thin stream upon a steam jet issuing at high pressure in a horizontal direction from an orifice, elongated horizontally so as to give a flattened formation to the jet. A suitable construction is specified.

Charcoal composition adapted for casehardening metals. F. C. ATKINSON. U. S. 1,361,131, Dec. 7. Charcoal is prepd. for use as a casehardening material by impregnating material such as wood chips, corn cobs or sawdust with $\text{Ba}(\text{OAc})_2$ soln., drying and charring.

Treating pickled metal plates. C. WILLIAMS. U. S. 1,360,843, Nov. 30. Plates of Fe or steel are treated with a hot alk. soapy soln. after removal from the pickling bath, washed, dried, formed into bundles and annealed. Cf. C. A. 14, 2334.

Protective treatment of iron blast furnace tuyères. SCHUSS GEBR. DAMPKESSEL-UND APPARATE-BAUANSTALT. Ger. 319,938, May 31, 1918. The castings are coated with a metallic or mineral prepn., or with a substance composed of fluorides, in order to protect them from the falling molten Fe. The coating may also be provided by converting the surface of the casting into the corresponding oxide by the action of O, or finally the coatings can be formed of a highly refractory enamel or by treatment with Zn or Cu. Any liquid Fe falling upon this surface flows off readily.

Cleaning hooks, needles, etc. J. GUSE. Brit. 151,688, June 19, 1919. Fish and other hooks, needles, pins, and like metallic articles, which require a high finish, are dipped in a soln. of $\alpha\text{-H}_2\text{BO}_3$, then immersed in a pickling bath of dild. HCl , swilled with H_2O , and scoured in a rotating barrel containing a soln. made up for 1 gal. of $\frac{1}{4}$ oz. cream of tartar, 8 oz. KOH or other neutralizing material, and 1 oz. Sn salts, and with or without a very small quantity of NH_3 . After scouring, the articles may be treated by the processes described in 145,116 (C. A. 14, 3063).

Apparatus and method of use for hardening manganese steel. F. SCHAEFFER. U. S. 1,362,147, Dec. 14. A cooling medium is supplied to a body of Mn steel under treatment (which has previously been heated to $1000\text{--}1100^\circ$) through nozzles fed from elec.-controlled valves. Cooling in progressive stages is employed and a uniform austenite product results.

Gages; measures of length. F. SMALLWOOD (trading as I. & D. Smallwood). Brit. 150,620, Feb. 7, 1920. Measuring rules and gages for length are, to avoid rusting and afford flexibility, constructed of duralumin, which is engraved by means of HCl or other acid.

Treating ores with air, chlorine or other gaseous reagents. C. S. FOGH. U. S. 1,362,408, Dec. 14. Mechanical features.

Detinning. W. C. ZACHARIAS. U. S. 1,362,381, Dec. 14. Sn is removed from coated articles by heating them to a temp. (preferably about 160°) at which the coating will be friable and then effecting mechanical disintegration and detachment of the Sn.

Furnace for melting aluminium or other soft metals. J. B. McCLAIN. U. S. 1,362,429, Dec. 14.

Furnace for annealing metals under non-oxidizing conditions. E. W. BEARDSLEY. U. S. 1,362,555, Dec. 14.

Furnace for heating metal rods or sheets. R. F. TALLEY. U. S. 1,362,369, Dec. 14.

Apparatus for continuous heat treatment of metal wire or sheets. R. E. TALLEY. U. S. 1,362,367-8, Dec. 14.

Apparatus for hardening and tempering wire in lead and oil baths. C. O. JOHN-SON. U. S. 1,362,321, Dec. 14.

Iron or steel directly from ore. L. P. BASSET. U. S. 1,360,711, Nov. 30. Fe or steel is directly made from ore by reducing the ore by the action of a carbonaceous material such as C and melting the metal produced by means of a very hot flame producing mainly CO and a minimum of CO₂ and H₂O vapor. Cf. C. A. 14, 925.

Making steel. A. H. HENDERSON. U. S. 1,361,333, Dec. 7. In producing steel in a furnace or converter, the metal is treated with 1% of a mixt. formed of chloride of lime 15, anhydrous Al₂O₃ 2 and anhydrous borax 2 parts in order to purify the metal. About 5 lbs. Pb per ton of steel may be added in the ladle to increase malleability and ductility. Cf. C. A. 14, 269.

Steel. STAHLWERKE. R. LINDENBURG AKT.-GES. Brit. 144,326, Aug. 6, 1916. Addition to 103,810 (C. A. 11, 1822). A steel alloy for use as a high-speed steel contains 0.5-0.8% of C, 6-10% of Mo, 3-6% of Cr, 0.2-0.4% of Mn, 0.2-0.4% of Si, and about 0.15 to 1% of V, with or without 0.5-2% of Co.

Steel. D. R. KNAPP. U. S. 1,361,589, Dec. 7. An initial charge of steel-forming materials is melted and refined and a small portion of the charge is then removed and replaced with a small portion of cold charging material. This mode of operation serves to promote continuity of production of metal ingots for rolling mills.

High-speed steel. W. L. TURNER. U. S. 1,360,830, Nov. 30. High-speed steel is produced by mixing a reducing metal such as Al with Fe and with an alloy metal compd. such as W oxide, the metal of which is capable of combining with Fe to form high-speed steel, heating the charge to reduce the alloy metal compd. and utilizing the heat evolved to melt the Fe and effect alloying with it of the reduced alloying metal.

Manufacture and heat treatment of high-speed steels. M. BERMAN. Ger. 310,042, Nov. 7, 1915. The W is replaced in whole or greater part by Cr. The steel contains Cr 7-12% and one or more of the constituents B, Mo, V, Ti, Co or W up to 1-2%. The steel of this compn. is shaped over-size, cooled, reheated and hammered to size. The steel is hardened by first heating to 50-100° above its hardening temp. of 700-800° and cooled in H₂O at 18°, and then hardened at the specified hardening temp.

Pig or cast iron from titanium iron sand or the like. INDUSTRIEELN MIJNBOW-MAATSCHAPP'G "TITAN." Ger. 319,561, Mar. 10, 1918. During the smelting of this ore with C in the elec. flame, resistance or induction furnace, undild. combustible gases (CO) are formed and conducted into a hearth or blast furnace in order to melt down the titaniferous Fe ore contained therein. The gas required for this fusion is obtained by melting down a portion of the titaniferous Fe ore with the reducing agent in the elec. furnace. Only so much gas is produced in the elec. furnace as is necessary

to melt down the charge in the blast furnace, while the additional heat required is generated in the blast furnace by the addition of coke. Cf. *C. A.* 12, 1457.

Armor plates of steel free from nickel. POLDHÜTTE, TIEGELGUSSTAHLFABRIK. Ger. 298,008, Oct. 17, 1915. The plates are made from a steel alloy which contains, besides the usual constituents, C 0.30–0.70%, Mn 0.30–1.30%, Si 1.40–2.30% and Cr 0.50–2.30%.

Heat-treating steel. M. T. LOTHROP. U. S. 1,360,551, Nov. 30. The machining properties of rolled steel are improved by hot working at a temp. above 1280° F.

Composition for tempering iron or steel. B. V. ALDRICH. U. S. 1,361,298, Dec. 7. A mixt. for tempering Fe or steel is formed of Na_2CO_3 6, S 4, black antimony 1, and rosin 1 part, mixed in pulverized condition.

Steel cement. W. R. CHAPIN. U. S. 1,362,472, Dec. 14. A mixt. adapted for use in cementing different steels is formed of ferrosilicon, borax glass powder and steel filings.

Alloys. C. B. BACKER. Brit. 151,400, July 10, 1919. See U. S. 1,341,774 (*C. A.* 14, 2160).

Alloys. FOSTER MILLIKEN. Can. 206,644, Dec. 21, 1920. An acid-resisting alloy contains Cu 50–60%, Ni 28–36, Zn 4–8, Fe 4–8.

Alloys. FOSTER MILLIKEN. Can. 206,645, Dec. 21, 1920. An acid-resisting alloy capable of withstanding high temp. contains Fe 16–20%, Cr 5–7, Cu 31–38, Ni 38–46 and Mn $\frac{1}{4}$ – $\frac{1}{2}$ %.

Alloys. F. MILLIKEN. Can. 206,729, Dec. 21, 1920. An alloy which resists high temp. and is easily machined contains Cu 56–64%, Ni 13–17, Zn 10–15, Fe 10–15.

Alloy. FOSTER MILLIKEN. Can. 206,730, Dec. 21, 1920. An alloy capable of being worked while hot contains Cu 40–48%, Ni 8–12, Zn 38–48, Fe 1–6, Mn 1–3.

Alloys. CALVIN VOS. Can. 206,713, Dec. 21, 1920. An alloy having degasifying and deoxidizing properties for treating steel contains Al 90–95%, Mg 2–10, NaF 0.01–5, UO_2 0.01–6 and FeSi 0.2–3%.

Iron-copper-nickel-zinc alloy. F. MILLIKEN. U. S. 1,360,773, Nov. 30. An alloy adapted for molds for casting glass is formed of Fe 8–12, Cu 55–65, Ni 12–18, Zn 11–17 and Si 0.5–1%.

Ternary alloy. LEON GUILLET. Can. 206,784, Dec. 21, 1920. An alloy contains Zn, Ni and 25–45% Cu, the % of Ni being greater as the percentage of Cu is smaller. 0.5 to 4.0% Pb may be added to make the alloy more easily worked.

Steel alloys. C. H. WILLS. Brit. 150,343, Aug. 24, 1920. Small proportions of Mo, up to 1% or a little over, are added to com. steels, which may contain also ordinary proportions of C, Si, S, P and Mn, with or without Cr or both Cr and Ni, the Cr being not more than 2% and the Ni not more than 5%. The products have improved physical properties, may be worked at higher temps. than usual, may be case hardened, and are susceptible to various heat treatments, sp. examples being quenched at 1500° F. and drawn at 1080–1100° F., or quenched at 1580° F. and drawn at 1040–1050° F.

Lead alloy. W. A. COWAN. U. S. 1,360,269, Nov. 30. Alloys consisting mainly of Pb are hardened by the addition of about 1–3% of Li. Li 1% gives an alloy of 17 Brinell hardness and Li 2% a hardness of 25. Hg 0.10–2.00% also increases the hardness.

Lead alloys. E. DE CAMPL. U. S. 1,360,272, Nov. 30. Alloys composed mainly of Pb, e. g., an alloy formed of Pb 97.88, Ba 1, Ca 0.50 and Hg 0.25 part are improved as to fluidity and tendency to dress by alloying with Al 0.02, Sn 0.25 and Zn 0.10%. The alloy is suitable for the manuf. of bearings.

Lead alloy. G. H. WORRALL. U. S. 1,360,346, Nov. 30. An alloy suitable for the manuf. of bearings is composed of Pb with Hg 1% or less and K 1% or less, with or without small amts. of Al, Sn or Zn.

Lead alloy. G. H. Worrall. U. S. 1,360,347, Nov. 30. A hard alloy suitable for making bearings is formed of Pb with Mg 2% or less and Hg 0.5%, with or without small amts. of Al, Sn or Zn.

Lead alloys. G. H. Worrall. U. S. 1,360,348, Nov. 30. The hardness of base lead alloys containing over 90% Pb, *e. g.*, alloys containing Pb and Ca or Ba or Sr or Na, K, Mg or Li 0.5%, is regulated by the addition of a small proportion of Hg which serves to increase the hardness of the alloys when added in an amt. of 0.25% of the alloy. Very small amts. of Al, Sn or Zn may be added.

Hard lead alloy. T. F. Wettstein. U. S. 1,360,339, Nov. 30. Hard alloys adapted for use in bearings are formed of Pb 97-99.25, Ba 2-0.50 and Ca 1-0.25 part.

Alloy for electrical contact points. L. T. Wallen and F. F. Wallen. U. S. 1,362,455, Dec. 14. An alloy for elec. contact points is formed of Ag 9 and Ni 1 part.

Corrosion-resisting ferrous alloys. Silas McClure. Can. 206,851, Dec. 21, 1920. An Fe alloy containing in excess of 0.5% Cu will withstand the action of the products of combustion of fuel. Cf. C. A. 14, 2160.

Flux for welding steel on iron. SÄCHSISCHE GUMMI- UND ASBEST-GESELLSCHAFT. M. B. H. Ger. 320,851, Sept. 26, 1917. SiC is intimately mixed with a powder composed of borax Fe filings and NH_4Cl .

Flux for welding steel on iron. SÄCHSISCHE GUMMI- UND ASBEST-GESELLSCHAFT. M. B. H. Ger. 322,415, Sept. 26, 1917. Addition to 320,851 (preceding pat.). The SiC specified in the principal patent may be substituted by Al_4C_3 .

Electrode for use in welding. R. Mattice. U. S. 1,361,269, Dec. 7. An electrode for welding is formed of a stranded cable including a central core strand of hard metal around which other strands of softer metal are twisted. Flux material is retained within the spaces between the strands.

Uniting by fusion. F. J. Heyes. Brit. 150,372, Apr. 2, 1919. In the elec. arc welding of Al, an Al electrode is used in conjunction with a flux of cryolite. The flux may be applied to the outside of the electrode in paste form preferably $1/16$ of an inch thick for a No. 8 S. W. G. electrode, and may be secured by an Al wire, thin sheet gauze, or a wrapping of cotton or other suitable fabric, or a binder may be incorporated in the paste. The flux may be placed in a hollow electrode of Al or in a series of transverse holes therein or in both. NaCl may be added to the cryolite for the more ready application and fixing of the flux and to cause the molten metal to flow more evenly by altering its surface tension. 15% of the NaCl may be added before mixing or the cryolite made into a paste with a 10% soln. of NaCl. NaCl may be replaced by NaHSO_4 . AlF_3 may be added to the flux to reduce the d. when molten. The parts to be welded are preferably pre-heated to a temp. between 300° and 400°. According to the Provisional Specifications the surfaces to be welded may be sprinkled with the powdered flux; also a satd. soln. of NaCl may be used in making the paste; also the pre-heating may be effected locally by a furnace or by alternating currents induced in the metal. Cf. 5, 138 (1900).

Rust-proofing ferrous metals. E. P. Andrews. U. S. 1,362,213, Dec. 14. A rust-resistant coating is formed on articles of ferrous metal by heating in an oxidizing atm. to a temp. of about 650° to form a coating of magnetic oxide and then immediately dipping in cottonseed oil or other oil.

Anti-rust protective coating for iron. H. Kozlisch. Ger. 319,855, Mar. 25, 1914. A nitrite or a substance containing nitrite is applied in the molten state to the surface of the article, or the latter is brought into contact with a solvent, emulsion or the like containing nitrite. *E. g.*, NaNO_2 50 g. is mixed with glycerol 2 kg., or NaNO_2 50 g. is mixed with vaseline 2 kg.

Producing an anti-rust coating of phosphate on iron. H. Arnold. Ger. 305,677, Sept. 17, 1916. The phosphoric acid or the phosphate soln. is always maintained at

the required degree of acidity. This can be effected by the addition of other acids such as H_2SO_4 or the like. The process may be shortened by employing solns. of higher concn. The soln. can be used as long as phosphate is present. The waste liquor, consisting of rather concd. FeSO_4 soln., may serve as a source of green vitriol.

10—ORGANIC CHEMISTRY

CHAS. A. ROUILLER

Influence of constitution on the rotation of optically active substances. XI. Normal and abnormal rotation dispersion and its calculation. H. RUPE AND ARTHUR AXERMANN. Univ. Basel. *Ann.* 420, 1-32 (1920); *J. Chem. Soc.* 118, 652; cf. *C. A.* 12, 147.—An examn. of about 50 dispersion curves leads R. and A. to the conclusion that the normal rotatory dispersion of org. substances can be expressed by the simple formula $\alpha_n = K/\lambda_n^2 - \lambda_0^2$ in which K is the "rotation const." and λ_0^2 is the dispersion const.; the expression has been used previously by Lowry and Dickson (*C. A.* 9, 3225). The formula is shown to be applicable to liquids in substance and in soln. It gives results which are in good harmony with the observed data over the visible spectrum and can probably be also employed outside these limits. In previous work, R. has attempted to distinguish between normal and abnormal rotatory dispersion on the basis of the displacement of the "characteristic wave length." It now appears that when a substance exhibits abnormal dispersion within the region of measurement, the characteristic wave length differs from the mean of the resp. class of compds.; but, although strong displacement is necessary for abnormal dispersion, it is not by itself sufficient. The method of calcg. the values of K_0 and λ_0^2 is fully discussed and it is shown that, provided the dispersion is normal, the calcd. values for the sp. rotations of a large number of compds. for a given wave length agree with the values observed within the limits of exptl. error. The following criteria of abnormal dispersion are then deduced, in which 3 types are distinguished: (1) Apparently normal rotatory dispersion with greatly displaced dispersion curve; the divergence between the calcd. and observed values does not amount to more than 3 times the mean exptl. error, but λ_0^2 and $\lambda\alpha$ differ markedly from the mean value for the appropriate class of compds.; (2) relatively abnormal rotatory dispersion, in which the differences are from 3 to 5 times as great as the mean exptl. error; and (3) total abnormal rotatory dispersion, in which the differences exceed 15 times the mean exptl. error. The connection between optical activity and constitution is discussed at length, and it is pointed out that comparisons between mol. rotations observed with a single wave length are inadequate for the elucidation of the problem, and that the rotatory dispersion must also be ascertained. The latter is greatly influenced by the solvent used and, at present, this action does not appear to be exerted in any regular manner even towards substances of the same type. The influence of constitution on K_m is described, and it is found that if the added radical is an aliphatic satd. hydrocarbon group and if the chem. change does not involve any considerable change in the optical properties of the asym. complex, K_m remains approx. const. in homologous series. The rule also applies when aromatic groups are introduced, provided that at least one $\text{CH}_2=$ group lies between the aromatic radical and the asym. complex. K_m is increased by the introduction of 1 or more ethylenic linkings, by a triple bond (the effect of this is noticeably less than that of 2 double bonds), and by the direct attachment of an aromatic nucleus to the asym. complex. Changes in K_m are also induced by one or more groups containing O. It thus appears that K_m is not affected by the number of groups required to form the mol., but is mainly dependent on the spatial distribution of the atoms and the relative distances from one another.

XII. Rotatory dispersion of the menthyl esters of certain keto acids. H. RUPE AND

H. KÄGI. *Ibid* 33-84.—This is an extension of the work of R. and Lenzing (C. A. 7, 3479), who prepd. a number of the esters, but observed their sp. rotation for the Na D-line only. The rotatory dispersions are now recorded. On the basis of this and other work 4 types of abnormal rotatory dispersion are distinguished: (1) Total anomaly of a mixture of 2 substances with opposed activities (Chugaev's extramol. anomalous dispersion); (2) total anomaly of a compd. containing 2 different asym. complexes, one of which is *d*-, the other *l*-rotatory (Chugaev's intramol. anomalous dispersion); (3) the rotatory dispersion curve does not pass through a max. or minimum, neither does it approx. to a horizontal line, but $\lambda\alpha$ and λ_0 differ widely from the normal values and $1/[\alpha]/\lambda^2$ gives bent or zig-zag lines (complex rotatory dispersion of Lowry and Dixon); (4) apparently normal course of the curves and $1/[\alpha]/\lambda_0^2$ gives straight lines, but $\lambda\alpha$ and λ_0 differ greatly (at least $\approx 15\mu\mu$ for the former) from the normal value for the particular class of compds. (relative anomaly). The following substances are described: (In the following, values are given for $[\alpha]$ and $[M]$ at 20° for C, D, Hg, and F lines.) Menthyl cyanoacetate, prepd. from KCN and $\text{ClCH}_2\text{CO}_2\text{C}_{10}\text{H}_{19}$, m. 83.5° , $[\alpha]$ —64.15°, —80.92°, —95.21°, —123.57°, $[M]$ —143.17°, —180.60°, —212.5°, —275.80°, in C_6H_6 . Menthyl phenylpropionate, $\text{PhC}(\text{CO}_2\text{C}_{10}\text{H}_{19})_2$, fine needles, m. $63-4^\circ$; d_{20}^{20} 0.8917; $[\alpha]$ —57.31°, —72.56°, —85.90°, —111.92°, $[M]$ —162.96°, —206.23°, —244.13°, —318.08°. Menthyl ethylacetacetate, d_{20}^{20} 0.9650; $[\alpha]$ —50.59°, —63.85°, —75.60°, —98.39°, $[M]$ —135.68°, —171.25°, —202.77°, —263.89°. Menthyl diethylacetacetate, $\text{C}_{15}\text{H}_{31}\text{O}_4$, formed by the action of Na upon a mixt. of 10 g. EtBr and 21 g. $\text{EtCHCO}_2\text{C}_{10}\text{H}_{19}$, b₁₆ 161°, d_{20}^{20} 0.9638; $[\alpha]$ —44.35°, —55.68°, —65.53°, —84.35°, $[M]$ —131.39°, —164.97°, —194.17°, —249.94°. In C_6H_6 the values are of the same order but smaller. Menthyl isopropylacetacetate, $\text{C}_{17}\text{H}_{33}\text{O}_4$, from 17 g. iso-PrBr and 25 g. dry Na salt of the menthyl ester in 50 cc. PhMe in a sealed tube at $120-125^\circ$ for 2.5 hrs., b₁₆ $153-9^\circ$, d_{20}^{20} 0.9603; $[\alpha]$ —48.69°, —61.53°, —72.91°, —95.05°, $[M]$ —137.39°, —173.65°, —205.76°, —268.24° in substance; for comparison the values in C_6H_6 are given: d_{20}^{20} 0.8855; $[\alpha]$ —47.32°, —59.74°, —70.58°, —91.70°, $[M]$ —133.53°, —168.59°, —199.19°, —258.79°. Ethyl diphenylmethylacetacetate, $\text{Ph}_2\text{CHCH}(\text{COMe})\text{CO}_2\text{Et}$, from the reaction of 2.3 g. Na in 35 cc. alc. upon 13 g. $\text{MeCOCH}_2\text{CO}_2\text{Et}$ and 24.7 g. Ph_2CHBr for 0.5 hr., small, glistening columns from alc., m. $89-90^\circ$. Menthyl diphenylmethylacetacetate could not be obtained from menthol and the Et ester or from menthyl acetacetate and Ph_2CHBr and alc. EtONa. Reaction could be caused between menthyl sodioacetacetate and Ph_2CHBr in dry C_6H_6 , giving a product which is resolved into diastereoisomerides by prolonged fractionation; *l*-menthyl *d*-diphenylmethylacetacetate, m. 118° , $[\alpha]$ —33.83°, —41.94°, —48.18°, —60.00°, $[M]$ —137.45°, —170.37°, —195.72°, —243.76°, in C_6H_6 (d_{20}^{20} 0.8944 or 10.31% soln.). *l*-Menthyl *l*-diphenylmethylacetacetate, m. 90° , $[\alpha]$ —51.13°, 65.15°, —78.04°, —103.94°, $[M]$ —207.73°, —264.68°, —317.15°, —422.30° (10% soln. in C_6H_6 , d_{20}^{20} 0.8918). *l*-Menthyl *dl*-diphenylmethylacetacetate, $[\alpha]$ —42.62°, —53.69°, —63.31°, —82.43°, $[M]$ —173.13°, —218.12°, —257.20°, —334.90°. The rotatory dispersion curves of the diastereoisomerides differ widely from one another and the *d* + *l*-form exhibits complex anomaly. Somewhat similar experiences are encountered with the benzylacetacetate esters; the most readily isolated ester is *l*-menthyl *l*-benzylacetacetate, $\text{AcCH}(\text{CH}_2\text{Ph})\text{CO}_2\text{C}_{10}\text{H}_{19}$, m. 68° , $[\alpha]$ —94.05°, —121.21°, —145.67°, —196.73°, $[M]$ —310.54°, —400.22°, —481.00°, —649.62° (in 10% C_6H_6 soln., d_{20}^{20} 0.8910). *l*-Menthyl *dl*-benzylacetacetate, m. $48-51^\circ$, $[\alpha]$ —43.43°, —55.10°, —65.20°, —85.85°, $[M]$ —143.41°, —181.95°, —215.30°, —283.48°. *l*-Menthyl benzylidenacetacetate (the hydrochloride of which m. 118°) has $[\alpha]$ —8.17°, —10.97°, —13.66°, —21.27°, $[M]$ —26.82°, —36.01°, —44.83°, —69.81° in C_6H_6 (10%, d_{20}^{20} 0.8932). *l*-Menthyl diacetylacetacetate, $\text{Ac}_2\text{CHCO}_2\text{C}_{10}\text{H}_{19}$, oil, b₁₆ $172-3^\circ$, d_{20}^{20} 1.0239; $[\alpha]$ —61.49°, —79.56°, —96.34°, —131.32°, $[M]$ —173.52°, —224.51°, —271.86°

—370.58°. In C_6H_6 (10%, d_4^{20} 0.8912) $[\alpha]$ —51.84°, —66.88°, —80.68°, —110.64°, $[M]$ —146.29°, —188.73°, —227.67°, —312.22°. Attempts to prep. *l*-menthyl benzoylacetate were unsuccessful. Menthyl benzoylacetate, (10% soln. in C_6H_6 , d_4^{20} 0.8930) $[\alpha]$ —50.95°, —64.39°, —76.15°, —99.89°, $[M]$ —153.98°, —194.60°, —230.13°, —301.87°. Menthyl benzoylbenzylideneacetate (10% soln. in C_6H_6 , d_4^{20} 0.8955) $[\alpha]$ —61.75°, —78.62°, —93.69°, —123.84°, $[M]$ —240.97°, —306.76°, —365.59°, —483.23°. Menthyl *l*-benzoylcinnamylacetate, (10% soln. in C_6H_6 , d_4^{20} 0.8945), $[\alpha]$ —67.41°, —86.08°, —102.18°, —135.71°, $[M]$ —281.97°, —360.06°, —427.40°, —567.68°. Menthyl dibenzoylacetate (10% soln. in C_6H_6 , d_4^{20} 0.8975) $[\alpha]$ —49.58°, —64.07°, —76.88°, —104.18°, $[M]$ —201.40°, —260.24°, —312.28°, —423.16°. Menthyl benzoylphenylacetate (10% soln. in C_6H_6 , d_4^{20} 0.8957) $[\alpha]$ 13.73°, 21.10°, 28.58°, 49.91°, $[M]$ 51.94°, 89.81°, 108.10°, 188.65°; in alc. with 1 drop C_6H_5N the values are (1.0845% soln., d_4^{20} 0.7983) $[\alpha]$ —43.89°, —58.91°, —75.08°, —93.57°, $[M]$ —166.01°, —222.80°, —283.96°, —353.88°. The d -rotation is caused by the activity of the asym. complex of the acid preponderating over that of the menthol; in alc., however the ketonic rapidly passes into the enolic form, thus destroying the asym. of the C atom in the acid portion of the mol. and leaving an activity entirely due to the presence of the menthyl group. α -Ester, $AcCH(CH_2CO_2Et)CO_2C_{10}H_{19}$, b_{D1} 111–2°, d_4^{20} 1.0295; $[\alpha]$ —44.76°, —56.67°, —67.24°, —87.97°, $[M]$ —146.00°, —184.85°, —219.32°, —286.94°; in 10% C_6H_6 soln., d_4^{20} 0.8028, $[\alpha]$ —45.59°, —67.80°, —87.99°, —99.38°, $[M]$ —148.70°, —188.53°, —221.78°, —291.56°. The β -ester b_{D1} 116–7°, d_4^{20} 1.0267; $[\alpha]$ —38.77°, —48.80°, —57.45°, —74.02°, $[M]$ —126.45°, —159.18°, —187.39°, —241.46°, while in a 10% C_6H_6 soln. (d_4^{20} 0.8016), $[\alpha]$ —40.94°, —51.59°, —60.68°, —78.29°, $[M]$ —133.54°, —168.30°, —197.93°, —255.38°. The product obtained, by heating menthol and Et acetylsuccinate is a mixt. of 90% α - with 10% of the β -ester. The enol content of several menthyl esters has been detd. by Meyer's method of titration with Br. The following %s. of enol were found, the figures for the corresponding Et esters being bracketed: Menthyl acetoacetate, 17.37 (7.71); menthyl ethylacetoacetate, 1.69 (3.1); menthyl diacetylacetate, 72.72 (90); menthyl acetylsuccinate 0.65 (3.7). Menthyl benzoyl- and phenylbenzoylacetates do not unite with Br.

C. J. WEST

Isomerism of the formylphenylacetates. V. Anilides and piperidides of formylphenylacetic acid. WILHELM WISLIZENUS AND RUDOLF ERBE. Univ. Tübingen. *Ann.* 421, 119–58(1920); cf. *C. A.* 11, 2323.—*Formylphenylacetanilide* (A), $C_{14}H_{13}O_2N$, is prepd. by the action of 90 g. HCO_2Et in 50 cc. EtOH upon a mixt. of 23 g. Na and 106 g. $PhCH_2CONHPh$ in 1.5 l. Et₂O. With vigorous stirring the soln. is complete in about 0.5 hr., and then the Na salt seps., which is completed in about 6 hrs. The salt is decompd. with H_2SO_4 . According to the choice of solvents, the α - or β -form may be obtained. Repeated crystn. from C_6H_6 or petr. ether gives the α -form, 6-sided plates, m. 68°. This is the labile isomer. It changes upon standing for several weeks to the β -form, more rapidly in a solvent or at 80°. The β -form is more easily obtained by crystn. from AcOH or Et₂O, in leaflets, m. 98°. The alc. soln. gives a deep violet color with $FeCl_3$. Crystn. from C_6H_6 or petr. ether gives the α -form. If the crude product is crystd. from EtOH, there results an *alcoholate*, $C_{14}H_{13}O_2N.EtOH$, fine aggregate of needles, m. 104–6° (the same product is obtained from either the α - or β -forms). The freshly prepd. soln. does not give the $FeCl_3$ reaction, but after 7–8 min. a violet color appears. The alc. is quickly lost in vacuum, less rapidly in the air. Heated with petr. ether for 6 hrs., the α -form resulted. The *menthyl alcoholate*, glistening needles, m. 107–8°, is somewhat more stable. Heated for 10 hrs. at 70° in vacuum, the β -form resulted. The α -form, crystd. from C_6H_6 , contained 96% enol form; the β -form, in alc., contained 77%, though this is not accurate due to the formation of the alcoholate. Both forms, after 16 hrs. in alc., contained about 6% enol. When shaken simultaneously with Br

and alc., the β -form contained 98.8% enolic form. The sodium compound could not be completely purified; it is sol. in hot alc., but does not cryst. FeCl_3 in alc. soln. gives a red color, $\text{Cu}(\text{OAc})_2$ a green ppt. The potassium compound is similar to the Na compd. The copper compound, $\text{Cu}(\text{C}_{13}\text{H}_{13}\text{O}_2\text{N})_2$, is prepd. by adding to 5 g. of the crude Na salt in 120 cc. Et_2O 3 g. $\text{Cu}(\text{OAc})_2$ in 50 cc. H_2O . Upon shaking a deep green salt seps., which crystals from alc. in fine glistening needles, m. $172-3^\circ$, or in prisms with 1 C_6H_6 from C_6H_6 . The iron compound, $\text{Fe}(\text{C}_{13}\text{H}_{13}\text{O}_2\text{N})_2$, was prepd. in a similar way, fine red needles, m. $207-9^\circ$. When A is heated to $120-140^\circ$, there is an evolution of CO_2 and β -anilido- α -phenylacrylic anilide results. Two mols. apparently react with the splitting off of CO_2 and PhCH_2CHO . β -Anilino- α -phenylacrylic anilide (B), $\text{PhNHCH}:\text{C}(\text{Ph})\text{CONHPh}$, from 7.2 g. of the α -form and an excess of PhNH_2 (2.5 mols.) by heating to 60° for 1 hr., flat 6-sided pale yellow crystals from AcMe , m. $107-8^\circ$. 3.6 g. A and 2 g. PhNHNH_2 in 40 cc. alc., boiled several hrs. under a reflux, gave the phenylhydrazone, $\text{C}_{21}\text{H}_{19}\text{ON}_3$, fine needles, m. $170-1^\circ$. When heated to $200-220^\circ$, PhNH_2 was split off, giving 1,4-diphenyl-5-pyrazolone (Ber. 20, 2932). PhNCO formed in 1 instance an addition product with A, $\text{C}_{22}\text{H}_{19}\text{O}_2\text{N}_2$, needles from acetone, m. $158-60^\circ$. In other expts. there was an evolution of gas, and B was formed. Conc'd. H_2SO_4 gave β -phenyl- α -carbostyryl (Ber. 28, 292), m. $227-8^\circ$. Upon oxidation of A or the alcoholate, there was isolated phenylglyoxylic anilide, m. $63-4^\circ$. Phenylacetopiperidine (C), $\text{C}_{13}\text{H}_{17}\text{ON}$, results by heating the $\text{C}_6\text{H}_{10}\text{NH}$ salt of $\text{PhCH}_2\text{CO}_2\text{H}$, long fine needles, m. $101-3^\circ$, at $180-200^\circ$. It was completely purified through the hydrochloride, small needles, m. $84-6^\circ$, and forms long needles, m. $5-7^\circ$, b₁₂ about $200-3^\circ$. Formylphenylacetopiperidine (D), $\text{HOCH}:\text{C}(\text{Ph})\text{CONC}_6\text{H}_{10}$, from 30.5 g. C and 16.6 g. HCO_2Et in 450 cc. abs. Et_2O and 4 g. Na, isolated as the sodium salt; the salt was decompd. with excess H_2SO_4 and crystd. from C_6H_6 -petr. ether, forming small crystals, m. $104-6^\circ$, and another portion, m. $110-6^\circ$. The higher m. form is probably impure β -form. If the Na salt is decompd. by passing CO_2 into the aq. soln., a hydrate results, $(\text{HO})_2\text{CHCHPhCONC}_6\text{H}_{10}$, short, fine needles from acetone, m. 114° . This does not give a FeCl_3 reaction, while the free base reacts with a violet color. The H_2O of hydration is given off after heating 8 hrs. at $70-80^\circ$, and the residue, crystd. from C_6H_6 , m. 122° . Methyl alcoholate, by twice crystg. the hydrate from MeOH , tables, m. $116-7^\circ$, which do not show a FeCl_3 reaction. Ethyl alcoholate, long prisms, m. 98° . By repeatedly crystg. the higher m. form, a substance m. $104-6^\circ$ was obtained (α -form?). Phenylhydrazones (E), $\text{PhNHN}:\text{CHCHPhCONC}_6\text{H}_{10}$, rectangular plates, m. $158-9^\circ$. Upon heating this splits off $\text{C}_6\text{H}_{10}\text{NH}$ and yields 1,4-diphenyl-5-pyrazolone. $\text{PhCH}_2\text{COCO}_2\text{Et}$ was obtained by the action of HCO_2Et upon $\text{PhCH}_2\text{CONPh}$. C. J. WEST

The diazo synthesis of aromatic arsonic acids and their theoretical importance in relation to similar reactions. An arsenic-containing by-product. HANS SCHMIDT. Heyden A. G., Radebeul at Dresden. Ann. 421, 159-74 (1920).—When PhNNO_2Na is added to a soln. of AsO_2Na_3 , there is evolution of N; earlier investigators considered this to be simply a reduction process: $\text{PhN}_2\text{ONa} + \text{AsO}_2\text{Na}_3 = \text{H}_2\text{O} + \text{C}_6\text{H}_6 + \text{N}_2 + \text{Na}_3\text{AsO}_4 + \text{NaOH}$. At the same time, however, there is a replacement of the diazo group by the As residue and there really results phenylarsonic acid: $\text{PhN}_2\text{Cl} + \text{AsO}_2\text{HK}_2 = \text{PhAs}(\text{O}_2\text{HK}) + \text{KCl} + \text{N}_2$. In carrying out the reaction a soln. of 198 g. As_2O_3 in 800 cc. 5 N KOH was dild. to 1 l. 50 cc. of this soln. was used for each 0.1 mol. diazo compd. In this way PhAsO_2H_2 , $1,2\text{-O}_2\text{NC}_6\text{H}_4\text{AsO}_2\text{H}_2$, $3\text{-O}_2\text{NC}_6\text{H}_3\text{AsO}_2\text{H}_2$, and $p\text{-AcNHC}_6\text{H}_4\text{AsO}_2\text{H}_2$ were prepd. In the case of PhAsO_2H_2 , there was obtained as a by-product, $\text{PhC}_6\text{H}_4\text{AsO}_2\text{H}_2$, previously described by Lettermann (Diss., Rostock, 1911), though the pure product does not m. 300° , while L. gives 275° .

C. J. WEST

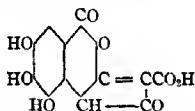
Aromatic antimony compounds. I. The diazo synthesis of aromatic stibinic acids and an investigation of their polymeric constitution. Aromatic derivatives of antimony

pentachloride and their rearrangement products. HANS SCHMIDT. *Ann.* 421, 174-246 (1920).—Phenylstibinic acid (*Ber.* 31, 2910), does not m. 285°. Depending upon the method of purification, the analysis seems to indicate the formula $(3\text{PhSbO}_2\text{H}_2\text{O}) \cdot 2\text{H}_2\text{O}$ or $(3\text{PhSbO}_2\text{H}_2\text{O}) \cdot 3\text{H}_2\text{O}$. Upon titration with alkali, only about $\frac{1}{2}$ mol. is required for neutralization. Upon standing the soln. becomes acid. This is explained by assuming that the pptd. phenylstibinic acid is a polymeric form, which in alk. soln. slowly is decompd. to a monomol. form. This monomol. form is stable only as the alkali salt, for upon acidifying, the free acid polymerizes again. This acid is not, therefore, a deriv. of antimonous acid, but of the polymeric form described by Delacroix (*Bull. soc. chim.* [3] 21, 1049). With H_2S the acid forms yellow sulfides. Concd. HCl gives a soln. of the tetrachloride, which gives a double salt with NH_4Cl , ammonium phenylchloroantimonate, $\text{PhSbCl}_4 \cdot \text{NH}_4$, pale yellow, voluminous powder, which does not m. 260°. H_2O decomp. it into PhSbO_2 and HCl . Even moist Et_2O decolorizes the salt. Other bases form ppts. with the tetrachloride (see *Ber.* 34, 2633; 36, 244, etc.). *m*-Nitrophenylstibinic acid may be expressed by the formula $[\text{O}_2\text{NC}_6\text{H}_4\text{SbO}_2 \cdot 2\text{H}_2\text{O}] \cdot 3\text{H}_2\text{O}$. The chloride, $\text{O}_2\text{NC}_6\text{H}_4\text{SbCl}_4 \cdot \text{H}_2\text{O}$, formed by dissolving the acid in concd. HCl , m. 140°. Ammonium salt, $\text{O}_2\text{NC}_6\text{H}_4\text{SbCl}_4 \cdot \text{NH}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$, sinters above 235°. *o*-Nitrophenylstibinic acid, $\text{C}_6\text{H}_4\text{O}_2\text{N}_2\text{Sb}_2$, light yellow-brown powder, not changed at 285°. The chloride, prepd. by dissolving in HCl - MeOH , leaflets, m. 127°. *p*-Chlorophenylstibinic acid, $[\text{ClC}_6\text{H}_4\text{SbO}_2 \cdot 2\text{H}_2\text{O}] \cdot 3\text{H}_2\text{O}$, dissolves easily in concd. HCl , and gives a yellow chloride, the ammonium salt of which forms citron-yellow crystals, $\text{ClC}_6\text{H}_4\text{SbCl}_4 \cdot \text{NH}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$, unchanged at 250°. *m*-Nitro-*p*-chlorophenylstibinic acid, prepd. by nitrating the *Cl* acid, nearly colorless powder, unchanged at 285°. The tetrachloride crystals, with 5 mols. H_2O , of which 4 are readily lost in vacuum. NH_4Cl gives a compound, $(\text{C}_6\text{H}_5(\text{NO}_2)\text{ClSbCl}_4)_2 \cdot \text{NH}_4 \cdot 8\text{H}_2\text{O}$, powder, m. 220° (decompn.). When the nitrochloro acid is warmed with KOH (1:1) on the water bath, the *Cl* is replaced by HO , giving *m*-nitro-*p*-hydroxyphenylstibinic acid, $[\text{O}_2\text{N}(\text{OH})\text{C}_6\text{H}_4\text{SbO}_2 \cdot 2\text{H}_2\text{O}] \cdot 5\text{H}_2\text{O}$, yellowish brown powder, which gradually darkens above 230°. The chloride is stable in the air, and yields a double salt with NH_4Cl . *p*-Anisylstibinic acid yields a chloride which decomp. very easily. II. The reduction products of aromatic stibinic acids, and *m*-aminophenylstibinic acid. The above described acids may be reduced to the trivalent Sb derivs., PhSbO , which are amorphous compds., forming characteristic halides. These oxides have a tendency to decomp., giving diaryl- or even triarylantimony compds. PhSbO , by reduction of the acid with $(\text{NH}_4)_2\text{S}$ in alc. HCl , voluminous electric powder, m. 153-4°. Iodide, PhSbI_2 , formed by reduction of the acid with SnCl_2 and then adding a satd. soln. of NaI , golden yellow needles, m. 69°. PhSbCl_2 , needles or tables, m. 62°. Reduction of the acid or oxide in AcOH -acetone with Na -bypophosphate at 65°, gave stibiobenzene, $\text{PhSb} \cdot \text{SbPh}$, brown powder, very readily oxidized. The course of the reduction depends upon the conditions of the expt., since other compds. with varying Sb content may be obtained. *m*-Aminophenylstibine chloride hydrochloride, *m*- $\text{HCl} \cdot \text{H}_2\text{NC}_6\text{H}_4\text{SbCl}_2$, by reduction of the NO_2 acid with SnCl_2 and crystg. from MeOH - HCl , needles, m. 218°. H_2O decomp. the chloride, giving $\text{Sb}(\text{OH})_3$ (see below). Iodide hydriodide, golden yellow, m. 160°. *m*-Aminophenylstibine oxide, $\text{H}_2\text{NC}_6\text{H}_4\text{SbO}$, by the action of alkalis on the chloride, amorphous powder, m. about 170°. Upon oxidation this yields *m*-aminophenylstibinic acid, $(3\text{H}_2\text{NC}_6\text{H}_4\text{SbO}_2 \cdot \text{H}_2\text{O}) \cdot 4\text{H}_2\text{O}$. The Na salt is easily sol. in H_2O . The acid may be diazotized and coupled with dyestuff components. *m*-Aminophenylstibine tetrachloride hydrochloride, *m*- $\text{H}_2\text{N}(\text{HCl})\text{C}_6\text{H}_4\text{SbCl}_4 \cdot 1.5\text{H}_2\text{O}$, crystals, m. 190°. The differences between these results and those of May (*C. A.* 7, 70) are probably due to the partial decompn. of M' 's products. Upon energetic reduction of the oxide, *m,m'*-diaminostibiobenzene, $(\text{H}_2\text{NC}_6\text{H}_4\text{Sb})_2$, is obtained, dark brown powder, which sinters 120°. *m*-Amino-*p*-chlorophenylstibine chloride hydrochloride, 3,4- $(\text{HCl} \cdot \text{H}_2\text{N})\text{ClC}_6\text{H}_3\text{SbCl}_2$,

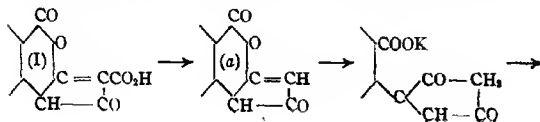
needles, m. 153°. III. Di- and triarylantimony compounds. $(\text{Ph}_2\text{Sb})_2\text{O}$, formed by heating the oxide in a boiling H_2O bath in a CO_2 atm. for 4 hrs., m. 78–80°. It also results when a soln. (5 g. in 50 cc. AcOH) is treated with 25 cc. 20% tartaric acid and allowed to stand; crystals of the tartrate (?), m. 132°, sep. HCl acting upon an AcOH soln. gave Ph_2SbCl , m. 68°. Oxidation of $(\text{Ph}_2\text{Sb})_2\text{O}$ with H_2O_2 or the action of PhN_2Cl upon PhSbO gave $\text{Ph}_2\text{SbO}_2\text{OH}$. This acid is sol. in NaOH , NH_4OH or Na_2CO_3 , and dil. warm HCl , but insol. in concd. HCl . This enables a sepn. of mono- and diantimonic compds., since the mono compds. are sol. in concd. HCl , and may be pptd. by NH_4Cl . *m*-Aminodiphenylstibinic acid, $\text{NH}_2\text{C}_6\text{H}_4\text{C}_6\text{H}_4\text{SbO}_2\text{OH}\cdot\text{H}_2\text{O}$, from PhN_2Cl and *m*- $\text{H}_2\text{NC}_6\text{H}_4\text{SbCl}_2\cdot\text{HCl}$, powder, sinters about 200°. The chloride darkens above 200°. *m,m'*-Diaminodiphenylstibine chloride hydrochloride, $(\text{H}_2\text{NC}_6\text{H}_4)_2\text{SbCl}_2$, by heating the mono- Ph deriv. in H_2O 1 hr., needles, m. 215°, easily sol. in H_2O , from which alkalis ppt. *m,m'*-diaminodiphenylstibine hydroxide, $(\text{H}_2\text{NC}_6\text{H}_4)_2\text{SbOH}$, which, when dried, sinters about 70°. Upon oxidation and subsequent treatment with HCl , *m,m'*-diaminodiphenylstibine chloride dihydrochloride, $(\text{H}_2\text{NC}_6\text{H}_4)_2\text{SbCl}_2\cdot 2\text{HCl}\cdot 2\text{H}_2\text{O}$, is formed, fine ppt., not changed at 250°. The aq. soln., treated with AcONa , ppts. *m,m'*-diaminodiphenylstibinic acid, $(\text{H}_2\text{NC}_6\text{H}_4)_2\text{SbO}_2\text{H}\cdot 0.5\text{H}_2\text{O}$, gray powder, decomps. 230°. Ph_3Sb is formed by heating PhSbO in a stream of CO_2 at 180–200°. In the same way $(\text{H}_2\text{NC}_6\text{H}_4)_2\text{Sb}$ is formed when the corresponding monoxide is heated at 120–5°. It is supposed, to account for discrepancy in properties, that this may occur in two forms, an amorphous, hydrated form, which sinters 80°, and a cryst. form from alc., m. 124°. The 2 differ in analytical figures by 0.5 H_2O . IV. Determination of antimony in antimony carbon compounds. 1. Aryl oxides and triarylstibines may, if an H_2N group is present in the C_6H_4 nucleus, be titrated directly with I in slightly acid soln. For other compds., the following procedure is used: 0.001 mol. is mixed with 0.2 g. NaCl and 3 g. NaHSO_4 in a Kjeldahl flask, and heated with 1.5 cc. HNO_3 and 10 cc. concd. H_2SO_4 1 hr. After cooling 1 g. $(\text{NH}_4)_2\text{SO}_4$ is added and the mixt. heated 0.5 hr., dild. to 300 cc., treated with 20 cc. 5 *N* HCl , reduced with H_2SO_3 and KBr and titrated with 0.1 *N* I.

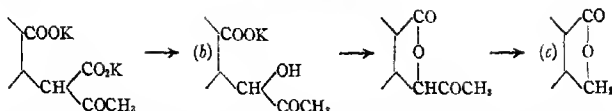
C. J. WEST

Galloflavin. J. HERZIG. Univ. Vienna. *Ann.* 421, 247–82 (1920); cf. *C. A.* 8, 1782 1104.—Trimethyloisogalloflavin has been decompd. to 3,4,5-trimethoxyphthalide, which suggests the following formula for isogalloflavin:



I. Decomposition of trimethyloisogalloflavin (with ELIZABETH FRYWELING and HERDWIG BRUNNER). Trimethyloisogalloflavin, upon distn., yields CO_2 and the compound (a) $\text{C}_{10}\text{H}_8\text{O}_5(\text{OMe})_3$, which does not react with CH_3N_3 or with the ordinary acetylating agents. Upon heating 1 g. with 25 cc. MeOH and 25 cc. 10% KOH for 30 min. in the H_2O -bath, then adding 25 cc. concd. HCl and again heating for 30 min., the compound (b) $\text{C}_{10}\text{H}_8\text{O}_5(\text{OMe})_3$, is formed, m. 74–77°; HCO_2K is split off during the process. When this is warmed on the H_2O bath with 10% KOH , AcOH is split off and trimethoxyphthalide (c) is formed. These changes may be represented as follows:





II. Action of potassium hydroxide and zinc dust upon isogalloflavin. (With FRANZ SCHAFFER.) 10 g. isogalloflavin and 100 g. Zn dust, intimately mixed, and 200 cc. 10% KOH were warmed on the H_2O bath for 3 hrs. and filtered hot into a mixt. of 100 g. H_2SO_4 and 100 g. H_2O , giving a 30-40% yield of a compound $C_{12}H_{10}O_7$, m. 191-4°. Methyl ether, $C_{13}H_{14}O_7(OMe)_2$, m. 101-3°. Ether acid, $C_{13}H_{14}O(OMe)_2(CO_2H)_2$, by boiling the ether with 5% MeOH-KOH soln. 1 hr., m. 200-3°. Upon distn. of the acid the compound $C_{13}H_{14}O(OMe)_2CO_2H$ is formed, m. 132-5°, which gives a liquid Me ester. Dimethyl ester, from $C_{12}H_{10}O_7$ and alc. HCl, m. 153-6°. Diacetyl dimethyl ester, $C_{16}H_{18}O(OAc)_2(CO_2Me)_2$, m. 71-3°. In addition to the C_{12} compd., a compound $C_{10}H_8O_4$ is formed, which is too unstable to be purified. Methyl ether, plates, m. 101-3°. Acid, $C_9H_8O(OMe)_2CO_2H$, by sapon. with alc. KOH, m. 185-8°. Monomethyl ester, $C_9H_8O(OH)_2CO_2Me$, by the action of 3% MeOH-HCl, m. 184-90°. Diacetate, $C_{11}H_{10}O(OAc)_2CO_2Me$, m. 109-12°. III. Attempts to transform $C_{12}H_{10}O_7$ to $C_{10}H_8O_4$. The action of Zn and KOH upon the compd. $C_{12}H_{10}O_7$ was studied, but it was not affected by these reagents. IV. Action of acetic anhydride, sodium acetate and zinc dust. (With KARL TRENNLE AND MARIANNE SCHLEIFFER.) A mixt. of 5 g. isogalloflavin, 5 g. AcONa and 15 g. Zn dust were boiled 3 hrs. with 100 g. Ac_2O , giving 5.6 g. of a product, m. 203-6° (unsharp), analyzing C 56.6, H 4.1, Ac 42.5%, probably $C_{11}H_{10}O_6Ac_4$. From trimethylisogalloflavin, a compound $C_{11}H_{12}O_6(OMe)_3Ac$ is obtained, m. 138-40°. Isogalloflavin methyl ester, m. 300-5°.

C. J. WEST

Several phthalide derivatives. J. HERZIG, HEOWIG BRUNNER AND MARIANNE SCHLEIFFER. *Ann.* 421, 283-92(1920).—3,4,5-Trimethoxyphthalide was prepd. according to Bargellini and Molina (*C. A.* 6, 3271). Upon oxidation to the *o*-phthalic acid, a m. p. of 145-8° was found, whereas the literature gave 174-6°. The methyl ester, $C_8H_7(OMe)_3(CO_2Me)_2$, m. 62-5°. 3,4,5-Trimethoxyphthalidecarboxylic acid, when heated with AcONa, Ac_2O and Zn dust, gave the phthalide. 5,6-Dimethoxyphthalidecarboxylic acid was obtained in very poor yield by hydrolysis of the condensation product of CCl_3CHO , H_2O and 2,3-(MeO) $_2C_6H_3CO_2Me$, m. 151-4°. Upon distn. in vacuum, meconin is formed, m. 99-101°. The action of Ac_2O , AcONa and Zn was studied in a preliminary way, but the reaction product is not meconin.

C. J. WEST

Aldol condensation and resin formation in the action of dilute alkali upon acetaldehyde. HARALD HAMMARSTEN. Stockholm. *Ann.* 421, 293-315(1920).—Aldol condensation is the principal reaction in the action of dil. alkali upon AcH. It occurs in as low a OH concn. as p_H 8.5 (a HO concn. of 2.5×10^{-4} g. ion per l.). The aldol does not disappear during the later reactions such as resin formation, even at higher temps. and longer reaction times. The first condensation product is acetal-dol, but this is changed in part to higher aldols, of which $MeCH(OH)CH_2CH(OH)CH_2CH(OH)CH_2CHO$ has been isolated. In no expt., under the conditions reported, was less than 40% of aldol obtained from 0.5 N AcH soln. This may be taken to indicate that the higher mol. aldols do not form aldehyde resins. Acetal-dol, however, does give rise to resins. Cannizzaro's reaction proceeds much lower with AcH than the aldol formation; in no case, even with the greatest variation in temp., times of reaction, concn., and condensation agents, did more than 12% of the AcH react according to this reaction. With the higher concns. of alkali the reaction was more pronounced, in relation to the aldol condensation, than with the lower concns., at least when KOH was the condensing agent. With $Ba(OH)_2$ the concn. was not so important. $Ph(OH)_2$ had a specific action, since a noticeable aldol condensation resulted but neither the Canniz-

zaro reaction nor resin formation. The Cannizzaro reaction occurs to about the same degree, other conditions being equal, whether one starts with 0.5 *N* AcH or 0.25 *N* aldol, so that it might be supposed that the reaction proceeded through the aldol step; this is not established, for it is possible that the alkali first decomps. the aldol to AcH, which then gives the Cannizzaro reaction. The aldehyde resin consisted of at least two constituents of different compn., the ordinary red aldehyde resin, investigated by Ekekrantz, and separable into α - and β -resins, and the light yellow, alk.-sol. resin. The latter appears to be formed if the alkali addition to the aldehyde soln. causes a clear yellow color, and is later made turbid by a yellow ppt., which, with increase in amt., becomes darker. Whether the red resin is formed directly or through the yellow resin is not established. The aldehyde resin formation is not a final reaction in the condensation of AcH; it is probable that acetate, EtOH, aldols, yellow and red resins are formed simultaneously. A preliminary investigation of the action of alkali upon acetone shows that an equil. between acetone and its condensation products is quickly reached, in which only about 14% of the acetone, at its b. p., is transformed. Diacetone alc. is presumed to be the principal condensation product, since this gives the same equil. products as acetone.

C. J. WEST

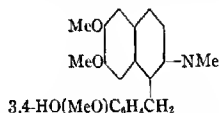
Mercuration. IV. Mercury derivatives of compounds with unsaturated linkings. W. MANCHOT, F. BÖSSENECKER AND F. MAHRLEIN. *Ann.* 421, 316-30(1920); cf. *C. A.* 14, 2926.—The mercuration of safrole with 1 mol. $\text{Hg}(\text{OAc})_2$ in aq. soln. proceeds at ordinary temp., and gave, upon the addition of NaCl, a well defined *chloride*, $\text{C}_{10}\text{H}_{10}\text{O}_3\text{HgCl}$, which corresponds to the addition of $\text{Hg}(\text{OH})\text{Cl}$ to safrole. With dil. HCl, safrole is easily split off from this compd. The chloride crysts. in long monoclinic prisms, m. 136-7°. The *bromide* forms long glistening needles, m. 144-5°. The *iodide* forms glistening needles, slightly yellow in color, m. 152.5°. Mercuration of eugenol methyl ether proceeds even more readily; the *chloride*, $\text{C}_{11}\text{H}_{10}\text{O}_3\text{HgCl}$, m. 112-5°. Eugenol itself did not yield a well-defined substance. Safrole does not, if instead of 1 mol. 3 mols. of $\text{Hg}(\text{OAc})_2$ are used. V. Mercuration of phenol ethers. W. MANCHOT AND F. BÖSSENECKER. *Ibid.* 331-345.—Volhard and Dimroth have shown that the mercuration of derivs. of C_6H_5 occurs in such a manner that a H atom attached to the ring is replaced by the group HgX . M.'s work on the mercuration of C_6H_4 and its derivs. has, however, rendered it not improbable that the primary products are additive compds. Expts. with various phenol ethers tend to show that such is actually the case. The products obtained with $\text{Hg}(\text{AcO})_2$ vary with the particular ether used, but may be classified as more or less completely hydrolyzed additive compds. They are readily decompd. by treatment with warm dil. HCl, and are immediately converted by alkali halides into products which are mercurated in the nucleus. *p*- $\text{MeC}_6\text{H}_4\text{OMe}$ reacts slowly with aq. $\text{Hg}(\text{OAc})_2$ at 50°, giving $3\text{C}_6\text{H}_4\text{Me}(\text{OMe})\text{Hg}(\text{OAc})_2 \cdot 2\text{HgO}$, thin colorless needles, m. 132-3°, immediately transformed by NaCl soln. into the product $\text{C}_6\text{H}_4\text{Me}(\text{OMe})\text{HgCl}$, needles, m. 162°. Anisole yields the compound $\text{C}_7\text{H}_7\text{OHg}(\text{OH})_2$, slender needles, m. 173°, which forms anisole when treated with dil. HCl, and *p*-anisylmercuric chloride, m. 236.7°, under the action of NaCl. Phenetole gives the substance $\text{EtOC}_6\text{H}_4\text{Hg}(\text{OH})_2$, small, matted needles, m. 135.5°, which is converted by KBr into *p*-phenylmercuric bromide, needles, m. 238°.

C. J. WEST

Synthesis of sinapine. ERNST SPÄTH. Univ. Vienna. *Monatsh.* 41, 271-85 (1920).—Sinapine is an alkaloid found in black mustard, and is the choline ester of 3,5-dimethoxy-4-hydroxycinnamic acid. Sinapic acid was synthesized by starting with trimethylgallic acid and passing through the following steps: 3,5-Dimethylgallic acid, carbethoxysyringic acid, its acid chloride, *carbethoxysyringic aldehyde*, $\text{C}_{12}\text{H}_{10}\text{O}_4$, m. 100-101°, *4-carbethoxy-3,5-dimethoxybenzalmalonic acid*, powder, m. 216-7°, *carbethoxysinapic acid*, m. 174°, sinapic acid. The acetylsinapic acid m. 188-93°, *acid chloride*, by the action of PCl_5 upon the Ac acid, m. 142-4°. *Hydroxyethylädimethylamine*

ester, $C_{15}H_{21}O_2N$, by condensing the acid chloride with the amine in H_2O or C_2H_5 , yellowish crystals, m. $127.5-8.5^\circ$. Treated in $CHCl_3$ with an excess of MeI , this forms sinapine iodide, identical with that prepd. from the natural alkaloid. It was further compared by changing it into the rhodanide. C. J. WEST

Constitution of laudanine. ERNST SPÄTH. Univ. Vienna. *Monatsh.* **41**, 297-304(1920).—The oxidation of Et laudanine gives 3-ethoxy-4-methoxybenzoic acid, m. $164-5^\circ$, which was synthesized from ethyl isovanillate, m. $51-2^\circ$, by the action of $EtONa$ and EtI , giving ethyl ethylisovanillate, m. 62° , and sapon. The oxidation of the carbethoxy deriv. of laudanine gave 3-carbethoxy-4-methoxybenzoic acid, m. $185-6^\circ$, which was also synthesized from isovanillic acid. These facts establish the constitution of laudanine to be:



C. J. WEST

Preparation of thiophosgene from thiocarbonyl tetrachloride. PERCY FRANKLAND, FREDERICK H. GARNER, FREDERICK CHALLENGER AND DOROTHY WEBSTER. *J. Soc. Chem. Ind.* **39**, 313-5T(1920).—The reduction of CCl_3SCl with Sn and HCl appears to be the most suitable method for the prepn. of $SCCl_2$. The addition of the $SCCl_4$ should be as rapid as is compatible with efficient condensation of the distillate. The ratio of $Sn:SCCl_4$ is best maintained at 0.5:1, which, while it gives a slightly lower yield of $SCCl_4$, gives a decidedly larger recovery of $SCCl_4$. Using 550 g. $SCCl_4$, 175 g. Sn , and 500 cc. HCl (28%), the yield of $SCCl_2$ was 53%, with a recovery of 38% unchanged $SCCl_4$. Steam may be used for heating the mixt. in place of direct heat. With Fe and $AcOH$, the yields of $SCCl_2$ were between 44 and 51%; with H_2S , 51%. 1 atom Cu gave 71% of $SCCl_2$. Zn dust gave unsatisfactory results. The fractionation of 2320 g. crude $SCCl_2$ (b. below 110°) gave 34 g. below 72° , 2063 g. between $72-76^\circ$, 84 g. from $76-110^\circ$. Fraction 2 had a $d_{15} 1.509$. C. J. WEST

Arsphenamine production by the Massachusetts Department of Public Health. HERMANN C. LYTGOE. *Chem. Age (N. Y.)* **28**, 390-1(1920).—This is a brief description, with no lab. details, of the method of manufg. arsphenamine, as carried out by the Mass. Dept. of Public Health during the war. The steps involved are: $PhNH_2 \rightarrow H_2NC_6H_4AsO_2H_2 \rightarrow AsO_2H_2C_6H_4NHCOC_2H_5 \rightarrow 1,2,4-C_6H_3(NH_2)(NO_2)AsO_2H_2 \rightarrow C_6H_3(OH)(NO_2)AsO_2H_2 \rightarrow$ arsphenamine. In a 2nd method the following intermediates were used: $PhOH \rightarrow p-HOC_6H_4AsO_2H_2 \rightarrow 1,2,4-C_6H_3(OH)(NO_2)AsO_2H_2$. C. J. WEST

Precautions in the use of dimethyl sulfate. MAX MUELLER. *Chem. Met. Eng.* **23**, 833(1920).—The poisonous action of Me_2SO_4 is due to its hydrolysis to $MeHSO_4$ and $MeOH$. The following precautions should be observed in its handling: All containers of Me_2SO_4 should be kept out of doors, but in a dry, cool place. Small bottles for lab. use should be kept in a separate room with other dangerous chemicals. Storage tanks for the plant should be installed in sheds outside the methylation building. The reaction tank should have a tight cover and a ventilating pipe leading through the roof, equipped with a steam injector so that a slight vacuum is formed in the pipe and any Me_2SO_4 vapor escaping is destroyed. All transferring of Me_2SO_4 from one tank to another should be done by vacuum. In cleaning app., the greatest amt. has to be removed; the remainder is destroyed by adding dil. $NaOH$ and heating with steam to 80° . Me_2SO_4 dropped on the floor should be covered with dry soda ash or soda lime and washed away with H_2O . Eyes, attacked with the vapors, should be washed with carron oil. Serious cases should be sent to the hospital. Clothes impregnated with

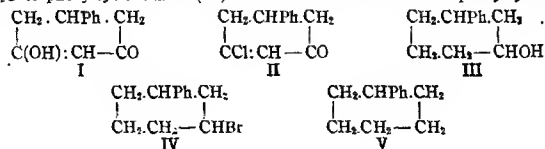
Me_2SO_4 should be removed immediately and washed with boiling H_2O and soda ash.

C. J. WEST

A new type of compound containing arsenic. GEORGE J. BURROWS AND EUSTACE E. TURNER. *J. Chem. Soc.* 117, 1373-80(1920).—Compds. of the type $\text{RR}'\text{R}''\text{As}$ combine with MeAsI_2 , PhAsI_2 , and EtAsI_2 to give the compds. $\text{RR}'\text{R}''\text{As.R}'''\text{AsI}_2$. These are completely dissociated in C_6H_6 but on evapn. form again. They ppt. with MeI the methiodide of the arsine. This additive reaction is not universal; thus Ph_2MeAs Ph_2As and other aromatic arsines would not combine with diiodoarsines under various conditions tried. *Phenyldiiodoarsine*, PhAsI_2 , was isolated for the first time; 34 g. PhAsCl_2 , 90 g. NaI and 100 cc. abs. EtOH are shaken 3 hrs., filtered, evapd. at low pressure to dryness, extd. with CHCl_3 , the CHCl_3 evapd. at low pressure and the product crystd. from EtOH in CO_2 snow. It forms lemon-yellow needles, m. 15° , b_p 190° (decompu.). PhMe_2As (A), PhEt_2As (B), PhMeEtAs (C), Ph_2MeAs (D), $\alpha\text{-C}_{10}\text{H}_7\text{Me}_2\text{As}$ (E), Ph_2As (F), $(o\text{-MeC}_6\text{H}_4)_2\text{As}$ (G), $(o\text{-MeC}_6\text{H}_4)_2\text{AsMe}$ (H), Me_2IAs (I), PhMeIAs (J), and PhMeClAs (K) were prepd. by new methods. A, from 19 g. PhBr , 2.9 g. Mg and 50 cc. Et_2O with 23.2 g. Me_2AsI in 50 cc. Et_2O let stand 2 hrs., treated with ice and dil. HCl , sepd. from the aq. layer, dried over Na_2SO_4 , evapd. and distd. at low pressure; yield 16 g. B, from 26.2 g. EtBr , 5.3 g. Mg and 40 cc. Et_2O , boiled 2 hrs. with 22.3 g. PhCl_2As in 100 cc. C_6H_6 ; yield 12 g. C, from 19 g. PhMeIAs in 50 cc. C_6H_6 , boiled 2 hrs. with 7.8 g. EtBr , 1.7 g. Mg and 20 cc. Et_2O ; yield 8 g. D, from 34.6 g. PhBr , 5.4 g. Mg and 90 cc. Et_2O with 34 g. MeIAs ; yield 17.5 g. E, from 50 g. $\alpha\text{-C}_{10}\text{H}_7\text{Br}$, 5.34 g. Mg and 200 cc. Et_2O boiled 1 hr. with 46.4 g. Me_2IAs ; yield 37 g. F, from 26 g. PhBr , 4 g. Mg and 50 cc. Et_2O boiled 0.5 hr. with 22 g. AsI_3 , removing all volatile compds. by heating at 200° under 12 mm. G, from 26 g. $o\text{-MeC}_6\text{H}_4\text{Br}$, 3.7 g. Mg and 80 cc. Et_2O with 22.8 g. AsI_3 ; yield 10 g. H, from 22 g. $o\text{-MeC}_6\text{H}_4\text{Br}$, 3.2 g. Mg , and 50 cc. Et_2O with 18 g. Me_2IAs in 100 cc. C_6H_6 ; yield, 8 g. I, prepd. by satg. with SO_2 , adding 1:1 HCl occasionally, 250 g. $\text{Me}_2\text{AsO}_2\text{H}$ and 800 g. KI in 1000 cc. H_2O (Me_2IAs seps. and finally free S), drying the oily layer over CaCl_2 , and distg. the product; yield 380 g. J, from 50 g. PhAsO in 30 g. NaOH in 240 cc. EtOH and 60 cc. H_2O , cooled and let stand overnight with 30 cc. MeI , acidified, freed from the EtOH by distn., treated with 50 g. KI and satd. SO_2 ; yield, 54 g.; NaOH gives better results than KOH . K, from PhMeIAs and the calcd. amt. of NaOH washed with H_2O , shaken repeatedly with small quantities of concd. HCl , and distd. The additive compounds were all prepd. by mixing equimol. wts. of the requisite arsine and haloarsine. $\text{PhMe}_2\text{As.MeAsI}_2$, lemon-yellow needles from $\text{Me}_2\text{CO-Et}_2\text{O}$, m. $93\text{--}4^\circ$, dissociates in C_6H_6 in concns. below 5%. $\text{PhMe}_2\text{As.EtAsI}_2$, yellow crystals from EtOH , m. 44° . $\text{PhMe}_2\text{As.PhAsI}_2$, orange prisms from EtOH or Me_2CO , m. 69° . $\text{PhMe}_2\text{As.PhAsCl}_2$, colorless needles from EtOH , m. 36° . PhEtAs.MeAsI_2 , bright yellow needles from EtOH or Me_2CO , m. $78\text{--}9^\circ$. PhMeEtAs.MeAsI_2 , yellow needles from EtOH , m. 84° . PhMeEtAs.PhAsI_2 , orange-yellow prisms from EtOH , m. 55° . $\text{C}_{10}\text{H}_7\text{Me}_2\text{As.MeAsI}_2$, yellow needles from EtOH , m. $76\text{--}7^\circ$.

C. C. DAVIS


Derivatives of phenyldihydroresorcinol. ALEXANDER J. BOYD, PERCY H. CLIFFORD AND MAURICE E. PROBERT. *J. Chem. Soc.* 117, 1383-90(1920).—Phenyldihydroresorcinol (I) with PCl_3 gives chlorophenylcyclohexenone (II) and this is reduced by Na in Et_2O to phenylcyclohexanol (III). HBr converts this to bromophenylcyclohexane



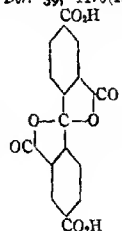
(IV) which may be reduced by Zn in H_2O -EtOH to phenylcyclohexane (V). Certain derivs. of I were prepd. Thus 5 g. of I, suspended in 50 cc. dry $CHCl_3$, with 4.3 g. of Br in $CHCl_3$ gives 4-bromophenyldihydroresorcinol, short glistening needles from EtOH, m. 177° (decompn.), sol. in EtOH, Me_2CO and AcOEt, slightly in boiling $CHCl_3$ and C_6H_6 , insol. in Et₂O and light petroleum. From 5 g. of I suspended in 125 cc. H_2O , treated with 4% $KMnO_4$ until permanently pink, and shaken for 5 hrs. is obtained 3.8 g. of a mixt., crystals from dil. HCl, of β -phenylglutaric and phenylsuccinic acids. 5-Chloro-1-phenyl- Δ^4 -cyclohexen-3-one (II), from 60 g. of I heated with 22 g. of PCl_5 and 240 g. of dry $CHCl_3$ for 3 hrs. on a water bath, freed from the $CHCl_3$ by distn., poured into cold H_2O , extd. 4 times with Et₂O, washed with 4% NaOH, then with H_2O , and dried over $CaCl_2$ (yield 65%), colorless plates from light petroleum, m. $63.5-64^\circ$, of a pungent odor, irritating to the skin, sol. in EtOH, Et₂O, $CHCl_3$, Me_2CO , C_6H_6 and AcOEt. Boiled with abs. EtOH and kept in moist air, it reverts to I in a few days. Oxime, short colorless needles from EtOH, m. 157° (decompn.). Semicarbazone, colorless needles from EtOH, m. 153.5° , decomp. 191° , insol. except in Et₂O. C_6H_6 , and boiling light petroleum. II (20 g.) suspended in 500 cc. H_2O treated with 4% $KMnO_4$ until pink for 20 hrs. gives 13.5 g. of a mixt. of phenylsuccinic acid, β -phenylglutaric acid and BzOH. II treated with twice the calcd. amt. of Na in Et₂O, washed with H_2O , dried over K_2CO_3 , evapd. and steam distd. until it gives no turbidity with K_2CO_3 , gives III, colorless needles (50-53% yield) from light petroleum, m. $79.5-80.5^\circ$, with an odor like geranium, sol. in ordinary org. solvents. A soln. in abs. EtOH with concd. H_2SO_4 gives a green-yellow color, changing to brown with green fluorescence. Ac deriv., clear oblong plates from EtOH, m. $43-4^\circ$, h. 300° . Bz deriv., colorless prisms from MeOH or EtOH, m. 68° , sol. in all org. solvents. o-Nitrobenzoyl derivative, from o- $C_6H_4(COCl)NO_2$ and the alc. in C_6H_5N , needles from MeOH or EtOH, m. 70° . IV, obtained nearly quant. by heating 5 g. of III 1 hr. with 25 cc. HBr (satd. at 0°) under pressure, is a colorless liquid, $b_{40} 186-7^\circ$, with an odor like geraniol. V, from 24 g. of IV in 75 cc. of 90% EtOH and enough abs. alc. to give a clear soln. heated 10 hrs. at 100° with 38.5 g. Zn dust mixed with sand (yield 74% after distg. over Na), colorless liquid, $b_{18} 233-4^\circ$, m. 6.5° . Phenylcyclohexan-3-one, from 14 g. of III and 126 g. of a mixt. of 60 g. $K_2Cr_2O_7$, 50 g. H_2SO_4 and 300 cc. H_2O kept at $50-55^\circ$ for 30 min. (yield quant.), colorless oil, $b_{18} 169-169.5^\circ$, $b_{176} 287-8^\circ$, with alc. H_2SO_4 gives a red-yellow color with green fluorescence. Semicarbazone, glistening prisms from EtOH, m. 187° , sol. in MeOH, EtOH, $CHCl_3$, AcOEt, Me_2CO , C_6H_6 , and boiling light petroleum, slightly in Et₂O. Oxime, colorless plates from EtOH, m. $128-9^\circ$, insol. in light petroleum, slightly in Et₂O, readily in other solvents.

C. C. DAVIS

Resolution of the keto-dilactone of benzophenone-2,4,2',4'-tetracarboxylic acid. WILLIAM H. MILLS AND CHARLES R. NODDER. J. Chem. Soc. 117, 1407-10(1920).—A

spirocyclic compd. of the type  should exist in 2 enantiomorphous forms.

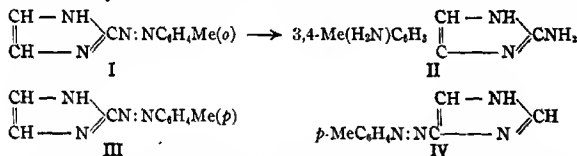
Previous attempts to prove the mol. asymmetry of such compds. by resolution into optically active antipodes have been unsuccessful (cf. Ber. 39, 1176(1906);

and C. A. 6, 1002, 2924; 7, 3330). The keto-dilactone (A)  of

{(2,4-HO₂C)₂C₆H₃CO (B) was prepd. as previously described (*Proc. Cambridge Phil. Soc.* 18, 149(1915)) and resolved into 2 optically active modifications with $[\alpha]_D^{20} = 17^\circ$ by an optically active base, PhCHMeNH₂ (C); 5.75 g. of B suspended in 100 cc. of MeOH was treated drop by drop with 4.09 g. C 12 cc. of MeOH, then with 110 cc. of dry. Et₂O. The product (7 g.), crystd. from a mixt. of 210 cc. of MeOH and 330 cc. of Et₂O, gave about 2 g. crystals which, decompd. with HCl (d. 1.08), washed with H₂O and dried, yielded a product, 1.0375 g. of which in 30 cc. of MeEtCO gave $[\alpha]_D^{20} = -18.3^\circ$ and 0.943 g. of which with 0.671 g. of *d*-base gave 1 g. of salt which decomp., yielding 0.6127 g. of acid. This in 131 cc. gave $[\alpha]_D^{22.5} = -16.9^\circ$. The highest specific rotation for the *l*-acid was $[\alpha]_D^{20} = -17.4^\circ$ in MeEtCO. The filtrate from the *d*-base *l*-acid salt on acidification gave 1.3 g. of acid of which 0.679 g. in 30 cc. of MeEtCO gave $[\alpha]_D^{21} = 7.9^\circ$. This was combined with *l*-PhCHMeNH₂ ($[\alpha]_D^{21} = -38.5^\circ$) by suspending 1.23 g. in 10 cc. MeOH, adding 0.87 g. of *l*-base in 2 cc. MeOH and pptg. with 6 cc. Et₂O. The liberated acid (0.6585 g.) was combined with 0.47 g. *l*-base. The salt gave on decompn. 0.569 g. of acid. This in 13.1 cc. MeEtCO gave $[\alpha]_D^{19} = 17.5^\circ$. From this acid by repeating the procedure 0.426 g. of acid was obtained which in 13.1 cc. MeEtCO gave $[\alpha]_D^{19} = 17.1^\circ$. The optically active forms of A are more sol. than the racemic in all solvents. When satd. solns. of the *d*- and *l*-forms in MeEtCO are mixed, a copious ppt. is formed. A soln. of the *d*-acid in 1% NaHCO₃ gave a rotation diminishing to 0.5 in 24 hrs. and disappearing in 4 days, showing the dependence of optical activity on the lactonic structure.

C. C. DAVIS

o- and *p*-Toluenazoglyoxalines. FRANK L. PYMAN AND LEONARD A. RAVALD. *J. Chem. Soc.* 117, 1426-9(1920).—*o*-MeC₆H₄N₂Cl combines with glyoxaline in Na₂CO₃ to form I in low yield, with considerable bis-*o*-toluenazo-*o*-cresol as by-product. With NaOH instead, a better yield of I was obtained but with different by-products, and with NaHCO₃ no I was formed.



Reduction of I gives II. *p*-MeC₆H₄N₂Cl with glyoxaline in Na₂CO₃ gives III in good yield with a small yield of IV and *p*-toluenazo-*p*-cresol as by-products. Reduction of III by SnCl₂ gives a 15% yield of 2-aminoglyoxaline with *p*-toluidine, guanidine and NH₃, but reduction by Zn + AcOH gives 97% *p*-toluidine and 42% glycocyanidine. I, prepd. by diazotizing 10.7 g. *o*-toluidine, adding to 6.8 g. glyoxaline, 20 g. Na₂CO₃ and 500 cc. H₂O at 5°, letting stand overnight, extg. with 5% HCl, adding Na₂CO₃ and crystg. from EtOH, brown-yellow crystals, m. 185-6°, sol. in EtOH and CHCl₃, slightly in Et₂O and C₆H₆. From 2 g. of I in 20 cc. hot 2.5% HCl + 12 cc. 40% SnCl₂, cooled and treated with 20 cc. concd. HCl is obtained 2,4'-diamino-4-*m*-tolylglyoxaline dihydrochloride in tiny needles, sol. in cold and hot H₂O. After drying it contains 1 H₂O. Addition of NH₄OH to its H₂O soln. forms a white ppt. darkening when filtered. Sulfate, woolly needles by addition of H₂SO₄ to its H₂O soln. Dipicrate seps. as a cryst. powder, m. 210°, sintering earlier, slightly sol. in boiling H₂O. III is similarly prepd. from *p*-toluidine. From EtOH, *p*-toluenazo-*p*-cresol was obtained. The HCl ext. made alk. with Na₂CO₃ yielded 15.2 g. of mixed toluenazoglyoxalines, m. 220°. Recrystn. from EtOH gave (1) 11.6 g. of 2-*p*-toluenazoglyoxaline (III), yellow leaflets from EtOH, m. 235°, sol. in boiling EtOH up to 5% (hydrochloride, cryst., deliquescent), and (2) 4-*p*-toluenazoglyoxaline (IV), yellow leaflets from EtOH, m. 152° (hydrochloride,

yellow needles from dil. HCl, m. 76°, loses 2 H₂O at 117°, remelts after darkening at 185°, very sol. in H₂O). C. C. DAVIS

The sulfonation of glyoxalines. FRANK LEE PYMAN AND LEONARD ALLAN RAVALLD. Univ. Manchester. *J. Chem. Soc.* 117, 1429-31(1920).—Direct sulfonation of glyoxalines has not been effected previously. Using acid containing 50 to 60% free SO₃ at 160°, an 83% yield of glyoxalinesulfonic acid was obtained. Properties of the acid itself, as well as those of its Na, NH₄, and Ba salts, are briefly noted.

J. T. R. ANDREWS

The catalytic action of iodine in sulfonation. JĀNANENDRA NATH RAY AND MANIK LAL DRY. Univ. Calcutta. *J. Chem. Soc.* 117, 1405-7(1920).—Ordinary H₂SO₄ and a trace of I can be used advantageously in place of the fuming acid, with or without the addition of P₂O₅. The nature of the product is changed in certain cases, thus making easy the prepn. of some acids otherwise difficult to obtain. Catalytic sulfonation is facilitated by the presence of an NH₂, HO, or halogen group in the nucleus, but proceeds with less ease in the case of CO₂H acids, and is inhibited in the case of NO₂ compds., thus explaining the non-formation of di-SO₃H acids in the product. It is noted that there is an optimum temp. for each reaction, with which max. transformation takes place. Some I vapor was noticed, but no traces of SO₂ or HI could be detected in the space above the reaction mixt.

J. T. R. ANDREWS

Chloro- α -bromocinnamic acids and their affinity for bromine. S. REICH, J. ARAUS, J. POTOK AND H. TEMPEL. *Helvetica Chim. Acta* 3, 793-800(1920).—A number of compds. are known to be unreactive toward Br even though they contain an ethylenic linkage. This inactivity has been attributed in some cases to the presence of such groups as NO₂, Ph, CO₂H and CN on the C atom α to the double bond. R. (C. A. 12, 1876) has shown that the mass, vol. of the group in question, the position in case of substituents in the C₆H₅ ring and the configuration of the mol. in the case of certain stereochem. isomers are the important factors. The authors measured the speed of the addition of Br to the 6 chloro- α -bromocinnamic acids, ClC₆H₄CH:CHBrCO₂H, when the Cl is in the *o*-, *m*- and *p*-positions, resp., in both *cis*- and *trans*-compds. The speed of the addition of Br to PhCH:CHCO₂H may be compared with that of the same reaction in the case of a compd. which has (1) a Br adjacent to a double bond and (2) a halogen in the C₆H₅ ring. The acids were prepd. for this work from *o*-, *m*-, and *p*-ClC₆H₄CHO by means of the Perkin reaction. The corresponding acids, m. 200°, 165°, and 245°, when treated in AcOH with Br, gave resp., *o*-, *m*-, and *p*-ClC₆H₄CHBrCHBrCO₂H, m. 181°, 176°, and 187°. These acids in alc. were then treated on the steam bath with 2 mols. of KOH for 2-3 hrs., most of the solvent was evapd., H₂O was then added and the acids pptd. with HCl. The *cis*- and *trans*-forms thus formed were sepd. by fractional pptn. *o*-Chloro- α -bromocinnamic acid (*trans*) crystd. from warm H₂O in needles m. 181°, very sol. in cold H₂O and in C₆H₆, insol. in ligroin, very sol. in alc. The methyl ester (by the HCl method) m. 51°; aniline salt m. 134°. The *cis*-form of the acid m. 128°, long needles from boiling ligroin and from warm H₂O, more sol. than the *trans*-acid in cold H₂O as well as in C₆H₆, insol. in cold ligroin, easily sol. in alc., Et₂O, CHCl₃, and glacial AcOH. In C₆H₆ the *cis*-form when exposed to sunlight was entirely transformed into the *trans*-form. The same thing happens when Br is added to the soln. in ligroin. The methyl ester m. 61°; aniline salt m. 123°. *m*-Chloro- α -bromocinnamic acid (*trans*) m. 140° and is sol. in warm H₂O, ligroin, and C₆H₆, easily sol. in alc. and glacial AcOH. The methyl ester m. 56°; aniline salt m. 116°. The *cis*-form m. 100° and is sol. in dil. AcOH but is insol. in ligroin, more sol. in cold H₂O than the *trans*-form. The methyl ester, needles, m. 53°; aniline salt, m. 77°. The *cis*-passes into the *trans*-form when the C₆H₅ soln. is exposed to the sunlight or is warmed to 220° for 0.5 hr., or when Br is added to the soln. in CS₂, or glacial AcOH and the mixt. is allowed to stand for 18 hrs. *p*-Chloro- α -bromocinnamic acid, m. 256°, is sol. in dil.

alc., insol. in H_2O , C_6H_6 , ligroin, petr. ether and CS_2 but easily sol. in alc., $CHCl_3$ and glacial $AcOH$. The methyl ester m. 82° . The *cis*-form m. 120° , is insol. in cold H_2O , petr. ether and easily sol. in alc., C_6H_6 , $CHCl_3$, and CS_2 , and goes over into the *trans*-form when the warm $CHCl_3$ soln. is treated with Br during 1 hr. The methyl ester m. 51° ; aniline salt m. 192° . Samples of the 6 acids finely powdered were weighed before and after being placed under a glass at the center of which was placed Br. The reaction proceeded in the dark at room temp. for 18 hrs. The excess of Br was pumped out and the increases in wt. measured. The conclusions drawn are that (1) the *trans*-modifications are the most stable and the least fusible and fix Br more slowly; (2) the position of Cl in the C_6H_4 ring has only a feeble influence on the speed of bromination in the *cis*-modifications. There is a slight augmentation in the speed in the order $m < p < o$; (3) in the case of the *trans*-forms a Cl atom in the *o*- or *m*-position prevents action of Br on the double bond. This does not hold when Cl is in the *p*-position.

H. E. WILLIAMS

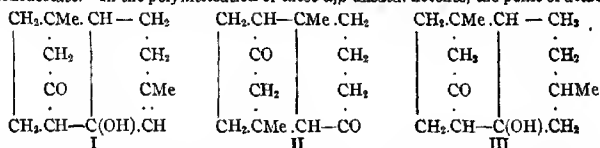
Polymerization of 1,2-cyclohexenone. L. RUZICKA. *Helvetica Chim. Acta* 3, 781-92(1920).—Certain 1,2-cyclohexenones polymerize to dimol. products under the influence of alkalis. Wallach (C. A. 2, 1830) mentioned in this regard $Me_3CHCH:CH:CH.CO.CH_2.CH_2$. Knoevenagel (*Ber.* 32, 423(1899)) transformed

dimethylcyclohexenone and phenylmethylcyclohexenone into cryst. dimol. products but could not transform the analogous compd. $Me_2C.CH_2.CMe:CH.CO.CH_2$. Fur-

ther references are wanting, due perhaps to the formation of noncryst. materials. Knoevenagel states that only 1 CO group is present in these compds. (oxime or phenylhydrazone). The second O atom cannot be characterized since there is no reaction with Ac_2O or $PhNCO$ and H_2O cannot be split out. While working on the polymerization of carvone, using $NaNH_2$, R. found that isophorone, $CH_2.CH_2.CH_2.CMe:CH.CO$

and $CH_3.CHMe.CH_2.CMe:CH.CO$ (A) undergo the same reaction, the product from

A being identical with that prepd. by Knoevenagel by the use of alkalis. These new products are liquids, boil unchanged and give monosemicarbazones. With Ac_2O monoacetates are formed and the second O atom is bound as an OH group. Bis-[1,3-methylcyclohexenone] (I) was reduced catalytically and the dihydro product gave a monoacetate. In the polymerization of these α,β -unsatd. ketones, the point of attack



is not at the CO group but at the ethylenic linkage. Rabc and his colleagues (*Ber.* 36, 225(1903); 37, 1667(1904), and C. A. 3, 1876) have worked out a scheme for the action of $AcCH_2CO_2Et$ on cyclohexenone and R. extends this scheme to the polymerization of 1,3-methylcyclohexenone with formation of an unsatd. ketone alc. on the one hand and a satd. diketone on the other. I agree well with all of the properties of bis-cyclohexenone. 1,2-Cyclohexenones with substituents in the 1-, 2- and 3-positions with respect to the CO group were investigated. Polymerization of the ketones proceeds more smoothly the greater the amt. of $NaNH_2$ used. At least 1 mol. of $NaNH_2$ should be used, otherwise some of the ketone remains unchanged. 1.5 mols. finely powdered $NaNH_2$ was added in small amts. at a time to the ketone in abs. Et_2O (4-5 parts), heated several hrs. under a reflux, cooled, treated with ice H_2O , filtered from a small

amt. of tarry material and the product dissolved in Et₂O and distd. *in vacuo*. After 1 distn. it is pure enough for analysis. A disemicarbazone of bis-1,2-cyclohexenone could not be formed from it even though the reagents were allowed to stand for 2-4 weeks. A diketone of the type II should give a disemicarbazone easily, as in the camphor and fenchone series, but a monosemicarbazone always resulted. *Bis-1,2-cyclohexenone acetate* was obtained by heating the polymer with 2 mols. Ac₂O in bomb tubes at 180° for 6-8 hours. 1,3-Methylcyclohexenone, $\text{CH}_2\text{CH}_2\text{CH}_2\text{CMe}:\text{CH.CO}$, when poly-

merized gave I, colorless oil *b*₁₂ 200-5°; takes up Br in CS₂ and gives off HBr. The *disemicarbazone*, m. 246° (decompn.), is difficultly sol. in alc. but sol. in glacial AcOH and concd. HCl. The easily sol. *semicarbazone* of I sepd. when the soln. was evapd.; it m. 215° (decompn.). The acetate of I *b*₁₂ 205°, *Dihydrobis-[1,3-methylcyclohexenone]* (III), from I in alc. with 10% Pt black and H₂, is a colorless oil *b*₁₂ 200°. The *semicarbazone* showed only a slight tendency to cryst. The *acetate* of III is a colorless oil *b*₁₂ 215°. A gave *1,5-dimethyl-1-cyclohexen-3-one*, m. 112° (*Ber.* 32, 423(1899)). The *acetate* was a sticky oil *b*₁₂ 200-2°. Isophorone gave *bisisophorone*, yellow oil *b*₁₂ 203-5°. The *semicarbazone* m. 215°; *acetate*, a sticky oil, *b*₁₂ 200°. There is no reference in the literature to the polymerization of carvone. It changes into carvacrol with strong acids and alkalis, and NaNH₂ does not act in this way. A sample of carvone which had stood for 10 yrs. was distd. to see if it had polymerized, due to long standing; 20 g. of an oily product *b*. 200-20° was obtained on distn. No cryst. semicarbazone could be obtained. The polymerization product, using NaNH₂, was *biscarvone* *b*₁₂ 225°, a yellow sticky mass. Crystals m. 118° were obtained. The *semicarbazone* m. 194-5°; *acetate*, a sticky product, *b*. 225°.

H. E. WILLIAMS

Synthesis of γ -piperidone. L. Ruzicka and V. Fornasir. *Helvetica Chim. Acta* 3, 806-18(1920).—The only unsubstituted keto compd. of the piperidine series, viz. α -piperidone (A), is not a true ketone but a δ -acid lactam. The isomeric compds. β - and γ -piperidones, may be considered true ketones. β -Keto compds. of piperidine are not mentioned in the literature. A few derivs. of γ -piperidone (C) are mentioned: Nortropinone (E) (*Ber.* 29, 393, 1578(1896)), triacetaminone and vinylidiacetonamine and analogs of the latter. These have substituents on C atoms adjacent to the NH group, and the compds. are stable. R. and F. sought to prep. C from γ -pyridone by catalytic reduction at room temp. Borsche (*C. A.* 9, 2093 (1915)) obtained in a similar manner tetrahydro- γ -pyrone from γ -pyrone. Hydroxypyridine could not be reduced by Pt and H in H₂O under 0.1 atm., but Emmert (*C. A.* 11, 1260) in glacial AcOH obtained γ -hydroxypiperidine (D). The reduction to the ketone only was not possible. *N*-Methyl- γ -pyridone could not be reduced catalytically to *N*-methyl- γ -piperidone. They sought to prep. C by oxidizing D with CrO₃, a method used for prepg. many derivs. of C. The method was not successful. C in the free condition condenses with itself and is only stable in the salt form whereas E dists. unchanged from alk. soln. with H₂O vapor. C may be sepd. from the original AcOH soln. of the oxidation products of hydroxypiperidine by handling the soln. with BzH and HCl. Petrenko-Kritchenko condensed a few aldehydes with CO(CH₂CO₂Et)₂ and NH₃ to form substituted ketopiperidinedicarboxylic esters (*J. prakt. Chem.* [2] 85, 1(1912)). R. and F. attempted to prep. the dicarboxylic ester of C and then by a ketone splitting to prep. C. CH₂O reacts in a different manner and neutral tarry products resulted. Following the condensation of aliphatic dicarboxylic esters by means of Na to cyclic ketone esters, they sought to prep. the carboxylic ester of C from β,β' -iminodipropionic ester (F) and thence by a ketone splitting to secure C. Heintz (*Ann.* 156, 41(1870)) prepd. F from MeCHICO₂H and NH₃. R. and F. did not consider the method satisfactory (Mulder, *Ber.* 9, 1904(1876)). F was prepd. from MeCHICO₂Et and MeCH(NH₂)CO₂Et. A side product was tertiary $\beta\beta'\beta''$ ammonia-tripropionic ester (K), N(CHMeCO₂Et)₃. The con-

densation of **F** with Na proceeds in the same way as that of adipic ester. The ketonic ester was not isolated but was heated directly with HCl and good yields of *dibenzal-γ-piperidone hydrochloride* (**G**) were obtained. When they attempted to get **C** from this product with NaOH solid odorless condensation products resulted. These seemed to be formed from 2 or more mols. of **C** with loss of H₂O. The stability of α, α' -derivs. of **C** depends on the hindrance caused by the presence of groups adjacent to the NH group. If the condensation product of **C** be warmed with HCl, then **G** may be prepd. from the product. This prepn. of **C** is the first use of the $\text{AcCH}_2\text{CO}_2\text{Et}$ condensation in the case of NH compds. R. and F. consider that this reaction can be used for prepg. other ketones in the piperidine series and they are extending the work in the case of pyrrolidone. Acetonedioxalic ester has been prepd. by Claissen (*Ber.* 24, 111(1894)) and the method has been modified by Willstätter and Pummerer (*Ber.* 37, 3745(1904); 38, 1465(1905)). R. and F. used 40 g. Na in 500 g. abs. alc. and to 0.5 of this soln. added a mixt. of 58 g. Me_2CO and 150 g. $(\text{CO}_2\text{Et})_2$, then 160 g. $(\text{CO}_2\text{Et})_2$; and the other half of the NaOEt, warmed the mixt. on the steam bath for 0.5 hr., distd. off half of the alc., cooled, added 1.5 times the calcd. amt. of 10% HCl, rubbed the ester in a mortar, and washed it with H₂O after filtering; yield 212 g. *N*-Methyl- γ -pyridone has been prepd. by Haitinger and Lieben (*Monatsh.* 6, 309(1885)) by the action of MeI and KOH on γ -pyridone. R. and F. used the procedure which Skraup used for prepg. *N*-methylutidone: 9.5 g. pyridone were heated with 30 cc. MeOH and 16 g. MeI in bombs at 140°. After distg. off the solvent the *N*-methyl- γ -pyridone-HI, m. about 250° (decompn.), was crystd. from Me_2CO . To secure the free base, the products remaining after the MeOH had been distd. off may be treated with KOH and the base may be extd. with CHCl_3 ; it is extremely hygroscopic and m. 86° in closed tubes. 6.2 g. γ -hydroxypiperidine (König and Neumann, *Ber.* 48, 956(1915)) was oxidized with 4.1 g. CrO_3 in 100 cc. AcOH, treated with 16 g. BzH satd. with HCl, freed after 2 days from the AcOH in a vacuum at 50° and from the excess of BzH with Et_2O , made alk. with KOH and then shaken with CHCl_3 . The product gave with dil. HCl **G**, yellow needles, m. 275-7° (decompn.). If the green soln. from the oxidation of γ -hydroxypiperidine be treated with KOH and extd. with Et_2O only unchanged γ -hydroxypiperidine results. **F** was prepd. not from the free β -aminopropionic ester (**H**) but from the HCl compd. 76.5 g. of the latter in 30 cc. alc. (abs.) were treated with the NaOEt from 11.5 g. Na, heated 8 hrs. with 114 g. $\text{CH}_3\text{ICH}_2\text{CO}_2\text{Et}$, treated with further amts. of NaOEt, using in all 2 mols., freed from alc. by distn., finally *in vacuo* at 50°, treated with solid KOH, using libe, extd. with Et_2O , dried with KOH and distd., giving 20 g. **H**, b. 110-120°, and a like amt. of **K**. 3 g. of **H** and 0.4 g. Na with 5 cc. xylene were refluxed and after a few min. a white product sepd. The reaction mixt. was finally poured into H₂O, treated with HCl, heated on the steam bath for several hrs., freed from the H₂O and xylene *in vacuo* and extd. with abs. alc. but no solid product could be obtained. Treatment with BzCl yielded no solid material. The residue after treatment with KOH and extn. with CHCl_3 yielded 0.6 g. oil having a piperidine odor and an alk. reaction to litmus and becoming thick on standing. The final product is insol. in Et_2O , difficultly sol. in H₂O, alc. and CHCl_3 , both hot and cold, does not yield a cryst. oxime nor benzoate. When heated with HCl it gave a residue from which **G** could be prepd. with BzH. 10 g. **H** with 1.5 mols. BzCl in C_6H_6 was heated until HCl ceased to be evolved, the excess BzCl removed by converting it into the ester with alc; the C_6H_6 soln. was shaken with Na_2CO_3 and the residue distd. *in vacuo*, giving benzoyl- β, β' -iminodipropionic ester, b. 195-205°.

H. E. WILLIAMS

Thiobenzoyl chloride. H. STAUDINGER AND JOS. SIEGWART. Zurich. *Helvetica Chim. Acta* 3, 824-33(1920).—*Thiobenzoyl chloride* (**A**), PbCSCl , was prepd. by passing Cl_2 through 80 g. SOCl_2 and 53 g. PbCS_2H (**B**) (cf. *Ber.* 39, 3224(1906)) in 50 cc. Et_2O .

at room temp., warming after 7 hrs. on a steam bath, removing the Et_2O and SOCl_2 , heating 0.5 hr. at 50° in abs. *vacuo*, raising the temp. of the oil bath to 150° when a decompn. occurs, and gradually raising the temp. further; the A b. $80\text{--}120^\circ$ (75% yield). After redistg. in *vacuo* 4–5 times, the pure A b. $60\text{--}5^\circ$; it is a deep violet, mobile liquid which was preserved several months in sealed tubes filled with CO_2 ; after a year the contents of the tube became viscous yielding unknown decompn. products. It has the properties of an acid chloride, forming with PhNH_2 , PhCSNHPH , m. 92° , with MeOH , PhCSOMe (C), b. $114\text{--}4.5^\circ$ (cf. C. A. 3, 2697), and with mercaptans dithiobenzoic acid esters (D). O decompd. A at $100\text{--}20^\circ$ into S and BzCl ; NaOH soln. decomp. A with formation of SO_2 ; C and D are quite stable to O_2 and NaOH ; Cl_2 and Br_2 decolorize A but the reaction products were not investigated; $\text{C}_2\text{O}_2\text{Cl}_2$ and SOCl_2 do not react further with A in the cold and $\text{Ph}_2\text{C}:\text{CO}$ does not react with it in cold Et_2O . When B and SOCl_2 are allowed to react slowly in Et_2O *thiobenzoyl trisulfide*, $[\text{PhC}(:\text{S})\text{S}]_3\text{S}$, often seps. in violet crystals on the sides of the flask; on longer standing these crystals liquefy and on distn. yield A. The S_2Cl_2 formed in the prepn. of A forms an *additive compound* (E) of the formula $[\text{PhCCl}(\text{SCl})\text{S}]_2$ or $(\text{PhCClS})_2$, which may also be obtained in the prepn. of A if the distn. is made under $15\text{--}20$ mm.; it is a red, viscous oil which gradually solidified; recrystd. from Et_2O , it m. $101\text{--}2^\circ$; on distn. under 1 mm. pressure A is obtained.

N. A. LANGE

The action of aliphatic diazo compounds upon thioketones. H. STAUBINGER AND JOS. SIGWART. Zurich. *Helvetica Chim. Acta* 3, 833–40(1920); cf. C. A. 1, 1286; 3, 2587; 10, 901; 14, 536.—Unlike the aldehydes, ketones are not known to react on the CO group with aliphatic diazo compds. Thioketones being more reactive were found to react as follows: $\text{Ph}_2\text{CS} + \text{Ph}_2\text{CN}_2 \longrightarrow \text{Ph}_2\text{C} - \text{S} - \text{N} = \text{N} - \text{CPh}_2$ (A)

$\longrightarrow \text{Ph}_2\text{C} - \underset{\text{S}}{\text{C}} \text{Ph}_2$ (B) $\longrightarrow \text{Ph}_2\text{C}:\text{CPh}_2 + \text{S}$. The reactivity of various substituted

aliphatic diazo compds. with Ph_2CS shows that non-carbonyl substituted compds., e. g., Ph_2CN_2 , $(\text{C}_6\text{H}_4\text{C}_6\text{H}_4)_2\text{CN}_2$ and PhCHN_2 react readily; the monocarbonyl substituted compds., e. g., $\text{N}_3\text{CHCO}_2\text{Et}$, $\text{PhBzCH}_2\text{N}_2$ react less readily whereas the dicarbonyl substituted compds., e. g., $\text{N}_3\text{C}(\text{CO}_2\text{Et})_2$ and $\text{Bz}(\text{EtO}_2\text{C})\text{CN}_2$, do not react. The nature of the thioketone also influences the reaction: highly colored aromatic compds. react readily while PhCS_2Et , PhCSNHPH and thiourea react very slowly if at all. Although it was to be expected that the highly colored dimethoxy- and tetramethyldiaminothiobenzophenone would be more reactive than Ph_2CS yet this was not found to be the case; it is thus seen that not in every case do auxochrome groups increase the depth of color and likewise increase the unsatd. character of the chromophore. *Tetraphenylethylene sulfide* (B) was prepd. by adding Ph_2CN_2 in petr. ether to a soln. of Ph_2CS (C) (cf. Ber. 29, 2944(1896)) until the blue color disappears, when B seps. in crystals, insol. in petr. ether, fairly sol. in Et_2O and readily sol. in CS_2 or CHCl_3 ; recrystd. from petr. ether + CHCl_3 it forms white crystals which soften 175° and m. 200° , decomp. quant. into $\text{Ph}_2\text{C}:\text{CPh}_2$ and S. *Diphenyldiphenyleneethylene sulfide*, prepd. in the same manner from C and $(\text{C}_6\text{H}_4)_2\text{CN}_2$ in Et_2O , seps. from CS_2 + petr. ether in white crystals which begin to soften 190° , then solidify and m. $220\text{--}5^\circ$, decomp. into $\text{Ph}_2\text{C}:\text{C}(\text{C}_6\text{H}_4)_2$, m. 225° . *p,p*-Dimethoxydiphenyldiphenylethylene sulfide, prepd. in PhH from $(p\text{-MeOC}_6\text{H}_4)_2\text{CS}$ and Ph_2CN_2 , seps. in white crystals on the addition of petr. ether, m. about 100° (decompn.). *p,p*-Tetramethyldiaminodiphenyldiphenylethylene sulfide was prepd. from $p\text{-Me}_2\text{NC}_6\text{H}_4)_2\text{CS}$ and Ph_2CN_2 ; after 2 days the soln. became colorless and the sepd. crystals were recrystd. from PhH, forming yellow needles, m. $164\text{--}5^\circ$; when heated to $210\text{--}20^\circ$ for 0.5 hr. it is decompd. into S and $(\text{Me}_2\text{NC}_6\text{H}_4)_2\text{C}:\text{C}(\text{C}_6\text{H}_4\text{NMe}_2)_2$, m. 208° .

N. A. LANGE

Ketenes. XXXIV. Diphenyl thioketene. H. STAUDINGER, G. RATESAM AND F. KJELSEBERG. Zurich. *Helvetica Chim. Acta* 3, 853-61(1920); cf. *C. A.* 14, 3423.—Diphenylthioketene (A) is very unstable, polymerizing immediately to a compound (B) of the type (a)...CPh₂CSCPh₂CS... or (b)...C(:CPh₂)SC(:CPh₂)S... of which (b) is the more likely. The polymerized A was found to react with PhNH₂ as follows: (Ph₂C:C:S)₂ (B) $\xrightarrow{\text{PhNH}_2}$ Ph₂CHC(:S)NHPH \longrightarrow Ph₂CHC(:NPh)NHPH-Ph₂C:CO (C). C reacts violently with P₂S₅ in CO₂ atm., forming a brown, resinous mass which contains some B; Ph₂CHCO₂Et and P₂S₅ react similarly at 100°, yielding almost the calcd. quantity of EtSH (D). Ph₂CHCOCl and D react in dil. NaOH, forming *ethyl diphenylthiolacetate*, Ph₂CHCOS₂Et (E), crystals from petr. ether, m. 50°. When E is heated with P₂S₅, A is not formed but D is obtained. C and CS₂ or PhNCS do not react at low temp. but CS₂ at 150° for 6 hrs. yields the polymer (F) of unknown constitution previously described (cf. Staudinger, *Die Ketene*, p. 39), m. 138-7°, and a small quantity of tetraphenylthioketocyclobutane (G), m. 244°; these are sepd. by recrystn. from EtOH in which F is more sol. PhNCS and C when heated at 100° for 5 days yield F and G. 3 g. Ph₂P:CPh₂ and 5 cc. CS₂ were heated to 60° for 16 hrs.; Ph₂PS (H) seps. on cooling; the CS₂ filtrate was evapd. and the residue on extn. with glacial AcOH, which removes Ph₂PS and resinous impurities, leaves B as a yellow amorphous material. Equimol. quantities of H and C in PhH were heated to 80° for 15 min. in an O-free atm.; on cooling 50% of unchanged H crystd.; Et₂O ppts. B from the filtrate; the mother liquors contain some B, H, Ph₂PO and other decompn. products not investigated. When H, C and PhH are heated in a sealed tube at 85°, CO is formed; petr. ether exts. Ph₂CS from the residue leaving H, B and compds. of unknown constitution as a residue. C (2 mols.) and H (1 mol.) in PhH were heated to 60° for 10 hrs.; Et₂O ppts. B, washed with glacial AcOH to remove a small quantity of Ph₂PO and unknown compds. Et₂PS and C react similarly. B is almost insol. in EtOH, Et₂O or glacial AcOH; it forms a colloidal soln. in PhH from which it is pptd. by Et₂O, EtOH or glacial AcOH. B, readily purified by repeated extn. with glacial AcOH, m. 262°, is an unusually stable compd., being unaffected by boiling EtOH, EtOH-KOH or PhNH₂ at low temp.; fusion with KOH causes decompn. while HNO₃ yields polymerized oxidation products. *Diphenylacetyldiphenylamidine*, m. 110°, was prepd. by heating B with an excess of PhNH₂ to 220-40° for several hrs. Equimol. quantities of PhNH₂ and B heated to 220-40° for 5 hrs. yield Ph₂CHCSNHPH.

N. A. LANGE

Ketenes. XXXV. The interaction of diphenylketene and thioketene. H. STAUDINGER. Zurich. *Helvetica Chim. Acta* 3, 862-5(1920); cf. previous abstract.—Tetramethyldiaminothiobenzophenone (A) reacts as follows: [Me₂NC₆H₄]₂CS + Ph₂C:CO \longrightarrow (I) [Me₂NC₆H₄]₂C—S—CO—CPh₂ \longrightarrow (II) [Me₂NC₆H₄]₂C:C—Ph₂ + (III) SCO. The intermediate ring compd. could not be isolated. Ph₂CS (B) yields an *addition compound* of unknown constitution for which the following are suggested: (IV) Ph₂C—CO—S—CPh₂; (V) Ph₂C—CO—CPh₂—S; (VI) Ph₂C=C—O—S—CPh₂; (VII) Ph₂C=C—O—CPh₂—S; although IV must be discarded as it does not yield SCO on heating. It was found that Ph₂C:CO (C) does not react with PhCS₂Et nor with PhCSCl. 16 g. C in petr. ether and 27.8 g. A in 200 cc. CHCl₃ shaken for 8 days, filtered and recrystd. from Me₂CO, yielded unchanged A and *asym-tetramethyldiaminotetraphenylethylene* (II), m. 111-2°. When B and C in Et₂O are allowed to react until the blue color disappears, the *addition compound* (IV, V, VI, or VII) seps. in 80-90% yield; it seps. from Me₂CO, CHCl₃, or C₆H₆Cl₆ in crystals, m. 180-1°, insol. in Et₂O or PhH. On melting it turns blue, de-

comp. quant. into B and C which recombine on cooling. If PhN:CHPh is added to the fusion, C is quant. converted into the lactam, m. 159–60°. The addition compd. does not react at room temp. with PhNH₂, acids or alkalis; when heated with PhNH₂, Ph₂CHCONHPh, B and Ph₂C:NPh are obtained; on slowly heating with aq. KOH, Ph₂CHCO₂H is formed. The addition compd. from (MeOC₆H₄)₂CS (D) and C prep. in Et₂O seps. and was recrystd. by dissolving in PhH and adding petr. ether: it is insol. in Et₂O or petr. ether and readily sol. in PhH or CHCl₃. A true m. p. was not obtained; it decompd. 80–120°; when heated in PhH, PhMe or C₆H₅Me, it dissociates into its components which recombine on cooling; Ph₂CHCONHPh and D are formed on heating the xylene soln. with PhNH₂.

N. A. LANGR

Naphthalenesulfonic acids. III. An alternative method for the qualitative detection of naphthalene-2,7- and -1,6-disulfonic acids. JOS. A. AMBLER. *J. Ind. Eng. Chem.* 12, 1194–5(1920); cf. *C. A.* 15, 55.—This is a confirmatory test for the microscopic method previously published. All of the naphthalene- β -, 1,5-, 2,6- and - α -sulfonic acids are removed as previously directed and the insol. salts from the Me₂CO extn. dried at 100° are treated as follows: They are boiled with 4–5 cc. EtOH-H₂O (4:1) and any insol. β -naphthylamine-2,6-disulfonate removed by filtration; on cooling the EtOH-H₂O soln. deposits the 2,7-(SO₃H)₂ acid. Another portion of the Me₂CO-insol. material is heated to boiling in H₂O, the β -C₁₀H₇NH₂ removed by titration with N NaOH and phenolphthalein, noting the vol. (a) of NaOH used, filtered and the filtrate evapd. to dryness, dissolved in a vol. of H₂O equal to 2a and an equal vol. of H₂SO₄ (d. 1.84) added; on cooling and rubbing with a stirring rod, the 1,6-(SO₃H)₂ acid ppts. while the 2,7-acid remains dissolved.

N. A. LANGR

The thermal decomposition of turpentine with particular reference to the production of toluene and isoprene. S. A. MAHOON. *J. Ind. Eng. Chem.* 12, 1152–5 (1920).—By passing turpentine vapors through electrically heated glass or Fe tubes, collecting separately the fractions which condensed below and above 50° and then subjecting these fractions to redistn., the following facts were established: With an empty glass tube the max. yield (4.5%) of isoprene (fraction b. 30–40°) was obtained at 600°; with a porcelain-filled glass tube 500° gave the best yield (4.4%) while an empty Fe tube produced the greatest yield (4.98%) at 450°. A mixt. of CuO and CuCl₂ or activated coconut shell charcoal in the tube gave no increase in yield of isoprene. Wood turpentine gave no greater yield than gum turpentine, and the yield was not increased by heating the turpentine at 250–70° before passing through the tube. PhMe was the chief constituent of the fraction b. 100–125°, while the fractions b. above 125° contained cymene, *m*-xylene and probably other *m*-comps. of the C₁₀H₈ series. The isoprene fraction gave with Br in Et₂O a substance with marked lachrymatory effect which, however, was lost on standing. Under the conditions used turpentine will not give sufficient yields of isoprene or PhMe to make it useful as a source of these comps., except perhaps in case of emergency. The article contains a review of work done on this subject and many references.

G. W. STRATTON

The acid hydrolysis of sugar-cane fiber and cottonseed hulls. E. C. SHERRARD AND G. W. BLANCO. *J. Ind. Eng. Chem.* 12, 1160–2(1920).—The digestion of bagasse containing 50 to 55% cellulose, with 1.8 to 2.5% H₂SO₄ at 100 to 125 lbs. pressure, gave 22.4 to 27.4% of reducing sugars of which 18.7 to 25.5% was fermentable, yielding 0.48 to 0.560% by wt. of EtOH. About 57% of the reducing sugars was xylose. Cottonseed hulls digested in a similar manner gave 9.6 to 16.7% of reducing sugars, practically none of which was fermentable. Sugar-cane fiber or bagasse are promising sources of xylose and furfural.

G. W. STRATTON

Pure phthalic anhydride. JULES BÉBIE. *J. Ind. Eng. Chem.* 13, 91–2(1921).—This note gives additional evidence to support the claim of Gibbs (*C. A.* 14, 3426) that phthalic anhydride, colorless needles m. above 130°, was manufd. previous to the

granting of the patent to Andrews (U. S. Pat. 1,336,182), which claims this compd. as an article of manuf.

G. W. STRATTON

The reduction of benzoic sulfonimide. F. GIANFORMAGGIO. Univ. Catania. *Gazz. chim. ital.* 50, I, 327-40 (1920).—Study of the products of reduction of aromatic acid compds. in alk. soln. has been limited to a few phenolcarboxylic acids and this work is here carefully reviewed. G. has chosen to study the reduction of the related compd. saccharin (A). Work on the reduction of amide and sulfonic compds. is scarce and is reviewed. No work on the reduction of A could be found. Nascent H (Na + AmOH) attacks the NH group of A first and eliminates it first as NH_3 . It then attacks the SO_2H group which may be liberated as SO_2 by subsequent acidification. Finally hexahydrobenzoic acid (B) is formed. The reactions are represented thus: (1) $\text{A} + 2\text{H}_2\text{O} \longrightarrow o\text{-HO}_2\text{SC}_6\text{H}_4\text{CO}_2\text{H}$ (C) + NH_3 ; (2) $\text{C} + 4\text{H}_2 \longrightarrow \text{B} + \text{H}_2\text{SO}_4$. A behaves like other aromatic amides having CO_2H in the ring so far as the NH group is concerned. The SO_2H group acts unusually. The S atom usually remains bound to the ring and is reduced to a sulfinic, sulfhydryl or mercaptan group. The previous results on the secondary reactions between AmOH and Na are reviewed. 50 g. pure AmOH + 10 g. Na were heated in a flask fitted with a dropping funnel and a reflux condenser in a bath at $145\text{--}50^\circ$ and 3.5 g. A in 100 cc. AmOH were added slowly from the dropping funnel, NH_3 is evolved almost immediately. 20 g. more Na are added a little at a time as required. When the reaction is complete, which requires several hrs., the mixt. is cooled to 125° and treated with 200 cc. cold distd. H_2O . After thorough agitation the alc. is sepd., and washed until no longer alk. The aq. layer and wash water are evapd. to $150\text{--}200^\circ$ and the soln. is acidified with HCl. This soln. extd. with Et_2O gives an ext. which on fractionation gives valeric acid b. 175° (from the AmOH) and hexahydrobenzoic acid, b. $232\text{--}3^\circ$. The former was identified by its Ag salt and the latter by its Ca salt.

E. J. WITZEMANN

Molecular organic compounds. IV. The addition of alkoxy to the nitro group. M. GIUA AND A. MARCELLINO. Univ. Sassari. *Gazz. chim. ital.* 50, I, 341-59 (1920).—The characteristic behavior of the NO_2 group in aromatic compds. (Giua, C. A. 10, 1858) has suggested the study of the behavior of definite groups toward NO_2 compds. In this paper results with aromatic compds. containing the alkoxy group are given. It is known that poly- NO_2 compds. give addition compds. with aromatic compds. containing phenolic OH. Thus tri- NO_2 derivs. of C_6H_6 , PhMe and PhOH combine chemically with aromatic phenols, giving more or less colored compds. It seemed of interest to know whether etherifying of OH in the phenol interfered with the reaction. The binary systems of $m\text{-(O}_2\text{N)}_2\text{C}_6\text{H}_4$, 2,4-($\text{O}_2\text{N)}_2\text{C}_6\text{H}_3\text{Me}$, α -trinitrotoluene and picric acid with $\beta\text{-C}_6\text{H}_4\text{OMe}$ and $p\text{-C}_6\text{H}_4(\text{OMe})_2$, resp., were studied cryoscopically and the results are given in tables. The results show that trinitrotoluene and picric acid give compds. with the 2 ethers but that the 2 other NO_2 compds. do not. The 2 ethers were selected in order to learn if the presence of additional OH groups influences the formation of addition compds. This is without influence except in the case of the compd. between picric acid and $\text{C}_6\text{H}_4(\text{OMe})_2$ which combine in the ratio of 2:3. The double ring in naphthol seemed to have no influence on the formation of addition compds. The mol. compds. that were observed here are all intensely yellow or orange-yellow. In this case as in various other similar cases the color of the compd. is in relation to the auxochrome group but which of the NO_2 groups is concerned is not yet known. On the basis of Werner's conception (C. A. 4, 456) it is necessary to admit a mutual satn. of the secondary valences in the chromophore and auxochrome group in mol. compds. and it is therefore not improbable that the addition compds. described here are formed by the exchange of valence lines between the characteristic functioning groups. From a comparative study of these and preceding results it is concluded that the substitution of the H with Me in phenolic OH diminishes their capacity to form addition

comps. Further generalizations cannot yet be made. For numerical details see the original.

E. J. WITZEMANN

The elements of the oxygen group and the organomagnesium compounds. M. GIUA AND F. CHERCHI. Univ. Sassari. *Gazz. chim. ital.* 50, I, 362-77(1920).—This paper represents the beginning of a study of the behavior of metalloid elements toward metalloorganic comps. and in this paper results with easily prepd. organomagnesium comps. are described. The literature on the behavior of organomagnesium comps. with O, S and Se is reviewed. No data on the behavior of Te with organomagnesium comps. could be found. Te reacts with PhMgBr as it does with S and Se to give first PhTeMgBr and then PhTeH , Ph_2Te and H_2Te . It was observed that the amt. of H_2Te formed is less than was expected on the basis of the reactions (1) $2\text{BrMgPhTe} \rightarrow \text{Ph}_2\text{Te} + (\text{BrMg})_2\text{Te}$; (2) $(\text{BrMg})_2\text{Te} + 2\text{HCl} \rightarrow \text{H}_2\text{Te} + 2\text{MgClBr}$. The fact that Ph_2Te is formed may be interpreted thus: $2\text{PhTeH} (+\text{O}) \rightarrow \text{PhTeTePh} (-\text{Te}) \rightarrow \text{Ph}_2\text{Te}$. Lederer (C. A. 9, 3235) treated Te halides with PhMgBr and obtained $(\text{PhTe})_2$ and PhTeH . G. and C.'s method is better. The authors have also examd. the reaction between S, Se and Te and pyrrole magnesium iodide (A). Angeli has recently shown that A reacts easily with O to give infusible black comps. but G. and C. were not interested in the reaction for that reason but because Madelung and Tencor (C. A. 9, 2253), treating Mg derivs. of indole and methylindole, obtained such comps. as diindolyl sulfide and dimethyldiindolyl sulfide. Under the conditions used only infusible blackish comps. were obtained which correspond to pyrrole black containing S, Se and Te, resp. These comps. were analyzed but no attempt was made to det. their constitution. The results show the perfect affinity in chem. behavior of the elements of the O group. The Te used was purified by fusing with KCN, the fusion mass was then taken up with H_2O , treated with a current of air and the Te filtered off, washed and dried. The expts. were done in a 200-cc. flask under a condenser fitted with a CaCl_2 tube. In the first expt. the materials were used in the ratio of 1 g. mol. to 1 g. atom Te and in the 2nd 2 g. mols. to 1 g. atom. 11.7 g. PhMgBr (from 1.6 g. Mg + 10.1 PhBr) in 50 cc. anhydrous Et_2O were treated in small portions with 8.2 g. powdered Te. After 12 hrs. at room temp. the mixt. was heated 8 hrs. but undissolved Te remained. The mixt. was treated with cold H_2O and then dil. HCl: some TeH_2 was evolved. 5.8 g. unchanged Te were recovered. The Et_2O ext. was a red-yellow, mobile, refractive oil. On fractionatio a colorless mobile fraction b. 70-80°, having the odor of C_6H_6 , and a fraction b. 120-30° were obtained. The 3rd fraction was an orange-yellow dense liquid with an odor like PhOH which did not dist. under 760 mm. without decompn. and constitutes the main product. The Et_2O soln. reacts with Br to give a yellow solid, m. 198-200° when crystd. from C_6H_6 . With aq. HgCl_2 it ppts. a yellowish white compd., m. 85-90° (decompn.), similar to that formed by L. (l. c.). With 10% aq. AuCl_3 a gray chloraurate is pptd., m. 154-6°. 10% H_2PtCl_6 soln. ppts. a yellow amorphous deriv., m. 75-80°. In a 2nd expt. 16.6 g. PhMgBr in 75 cc. Et_2O were treated with 5.8 g. Te and the mixt. was kept 86 hrs. at room temp. and heated 68 hrs. The Et_2O ext. was fractionated under 85 mm.: (1) Up to 180°, colorless refractive, mobile liquid; (2) 130-55° (mostly 150-5°), dense bright yellow liquid which solidifies in part; (3) 155-200°, dense yellow liquid which mostly solidifies to give colorless scales (m. 84°); a dense red residue with a disagreeable odor from which some Te seps. on standing, remained. A part of fraction 4 was treated with HNO_3 (d. 1.40) in excess and then thrown into much H_2O . The yellow-red ppt. crystd. from EtOH , m. 210-2° (decompn.), and is the nitrate of phenyltellurinic acid, PhO_2TeN . The EtOH mother liquors on evapn. gave shining white crystals that m. 66-8° to a colorless liquid, solidify and m. again at 201-3° (decompn.). This is apparently due to loss of EtOH of crystn. A portion of fraction 4 treated with Br in Et_2O gives yellow crystals, m. 203°, of what may be a diphenyltellurium bromide. EtMgI from 2.4 g.

Mg + 16 g. EtI in 60 cc. Et₂O were treated with 7 g. pyrrole in 10 cc. Et₂O drop by drop, heated on a H₂O bath 1 hr. and then treated with 3.2 g. powdered S in small amts. The mixt. browns and seps. a voluminous black substance. The solid part and the Et₂O soln. were examd. separately and are described. Similar expts. with Se and Te are described.

E. J. WITZEMANN

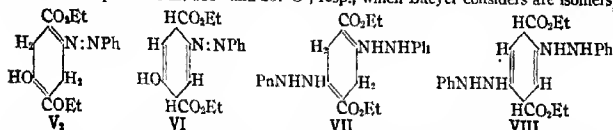
Molecular compounds of sulfurous anhydride with the amines. A. KOREZYNSKI AND M. GLEBOCKA. Univ. Poznan. *Gazz. chim. ital.* 50, I, 378-87 (1920).—The app. for the absorption of the SO₂ was that previously used (*C. A.* 3, 653). The compds. used were as dry as possible and the absorption was carried out at 0°. In each case the SO₂ was detd. In the following the name of the compd. used is followed by the formula of the product as deduced from the SO₂ detns.: *p*-Toluidine, C₇H₇NH₂.SO₂, canary-yellow; α -naphthylamine, C₁₀H₇NH₂.SO₂, orange-yellow; *p*-chloroaniline, C₆H₄ClNH₂.SO₂, canary-yellow; *p*-bromoaniline gave C₆H₄BrNH₂.0.5SO₂ at room temp. and C₆H₄BrNH₂.SO₂, canary-yellow, at 0°; 3,5- and 2,4-dichloroaniline gave C₆H₃Cl₂NH₂.SO₂, canary-yellow; 3,5-dibromoaniline, C₆H₃Br₂NH₂.SO₂, canary-yellow; *p*-nitrosodimethylaniline, C₆H₅N₂O₂.2SO₂; diphenylamine, C₁₂H₁₀NH.0.5SO₂, orange-yellow; γ -chloroquinoline, C₁₀H₇ClN.1.5SO₂; aminoazobenzene, C₁₂H₉N₂NH₂.SO₂; dimethylaminoazobenzene, PhN₂C₆H₄NMe₂.0.5SO₂; dimethylaminoazobenzene acid, Me₂NC₆H₄N₂C₆H₄CO₂H.SO₂; thiourea, CSN₂H₄.0.5SO₂, colorless; ethyl *p*-aminobenzoate, H₂N-C₆H₄-CO₂Et.0.5SO₂, canary-yellow; its propyl ester does not unite with SO₂; *o*-aminocinnamic acid, C₉H₇O₂NH₂.SO₂, bright yellow; 2,4-diaminotoluene, MeC₆H₃(NH₂)₂.SO₂, canary-yellow; *p*-nitro-3,5-phenylenediamine, C₆H₃NO₂(NH₂)₂.0.5SO₂, yellow; benzidine, C₁₂H₈(NH₂)₂.2SO₂, canary-yellow; 1,3-diaminoazobenzene, C₆H₅N₂C₆H₄(NH₂)₂.0.5SO₂; tetramethyldiaminotriphenylmethane, C₂₂H₂₄N₂.2.5SO₂; leucoaniline, C₉H₇N₃.3SO₂, yellow; *p*-leucoaniline C₁₀H₉N₃.1.5SO₂, yellow; rosaniline, C₂₀H₂₁ON₃.1.5SO₂; *p*-rosaniline, C₁₉H₁₉ON₃.SO₂; antipyrine, atropine, cocaine and narcotine give liquid addition products; dimethylaminoantipyrine, C₁₃H₁₇ON₃.SO₂, canary-yellow; hexamethylenetetramine does not give a definite mol. compd.; caffeine, C₈H₁₀N₄O₂.0.5SO₂, colorless; strychnine, C₂₁H₂₂O₄N₂.2SO₂, clear yellow; brucine, C₂₈H₂₈N₂O₄.3SO₂, yellow; morphine, C₁₇H₁₉NO₃.1.5SO₂, canary-yellow; quinine, C₂₀H₂₄N₂O₂.SO₂, lemon-yellow; papaverine, C₂₀H₂₁NO₄.SO₂, straw-yellow; veratrine, C₂₂H₂₅NO₃.2.5SO₂, yellow; hematin gives a complex having nearly 3 mols. SO₂ in it; hemin does not unite with SO₂. 31 other compds. which are not mentioned above were tried but failed to give addition compds. No difference was observed in the behavior of primary, secondary or tertiary amines in their ability to combine with SO₂. The colorless amines that contain an aromatic nucleus give yellow products while those without this nucleus remain colorless. The appearance of color indicates that mol. compds. are formed and that the complex is the cause of the color. The color being the same with all classes of amines indicates that the secondary valences are involved. The action of the secondary valences of the N atom is paralyzed and destroyed by negative substituents (OH, CO₂H, SO₂H, CO, CS and NO₂ as well as halogens). The influence of OH and NO₂ is greater than that of halogens. No direct relation between the number of NH₂ groups and the SO₂ bound could be established. The behavior of CO₂, H₂S, NO and other organic compds. is being studied.

E. J. WITZEMANN

Unsaturated compounds in organic chemistry. II. The constitution of succinylsuccinic ester. M. GIUA. Univ. Sassari. *Gazz. chim. ital.* 50, I, 387-93 (1920).—The esters of succinylsuccinic acid (A) have been assigned a variety of formulas of which the following are the principal ones: (I) $\text{RO}_2\text{C}.\text{CH}.\text{CO}.\text{CH}_2.\text{CH}(\text{CO}_2\text{R}).\text{CO}.\text{CH}_2$;

(II) $\text{RO}_2\text{C}.\text{C}(\text{OH}).\text{CH}_2.\text{C}(\text{CO}_2\text{R}).\text{C}(\text{OH}).\text{CH}_2$; (III) $\text{RO}_2\text{CCH}.\text{CO}.\text{CH}_2.\text{CH}_2.\text{CO}.\text{CH}.\text{CO}_2\text{R}$; (VI) $\text{RO}_2\text{CCH}.\text{CO}.\text{CH}_2.\text{CH}(\text{CO}_2\text{R}).\text{CO}.\text{CH}_2$. Of these I and II are generally

admitted (cf. Meyer and Kappelmeier, *C. A.* 6, 88; Hantzsch, *C. A.* 9, 2095). G. has studied the action of PhNHNH_2 on A which has already been studied by Knorr (*Ber.* 17, 2053(1884), Baeyer, (*Ber.* 24, 2692(1891)) and Kishner (*Ber.* 26, 590(1893)), who isolated products of uncertain constitution. The products may be phenylhydrazones (CO reacting), benzenedihydrazo compds. (enol reacting) or pyrazole derivs. The constitution of the products obtained differs depending on whether AcOH was present in the reaction mixt. of $\text{PhNHNH}_2 + \text{A}$. G. has allowed A to react with PhNHNH_2 and PhMeNNH_2 in EtOH in the mol. ratio 1:1 and 1:2 in the presence of AcOH. When they react in the ratio of 1:1 the main product is a red substance, m. 142° , that is certainly an azo compd. having the formula V or VI. When they react in the ratio 1:2 the main products m. 160° and $207-8^\circ$, resp., which Baeyer considers are isomers,



(VII) and (VIII), of benzenedihydrazodihydroterephthalic acid. Baeyer found that by treating the compd. m. 166° with $\text{Cu}(\text{AcO})_2$ in hot EtOH benzaldiazodihydroterephthalic acid is formed. A tar was also obtained which, distd. under 52 mm., decomps. at above 100° , giving an oily liquid containing PhNH_2 . With PhMeNNH_2 A gave only derivs. like V and VI even when the ratio 1:2 was used.

E. J. WITZEMANN

The reactions of some *o*- and *p*-substituted derivatives of benzene. A. ANGELI. *Gazz. chim. ital.* 50, II, 1-8(1920).—Some years ago A. discussed (*C. A.* 12, 365) the analogy in the behavior of H_2O_2 , NH_2OH and NH_2NH_2 with that of $p\text{-C}_6\text{H}_4(\text{OH})_2$, $p\text{-C}_6\text{H}_4(\text{OH})\text{NH}_2$ and $p\text{-C}_6\text{H}_4(\text{NH}_2)_2$ and said that in some reactions the latter compds. behave as though the substituents were joined directly as in the 1st group. In this paper A. discusses other transformations which extend and confirm this relation. RMe (R = a negative group) condenses easily with esters thus: $\text{RCOMe} + (\text{CO}_2\text{Et})_2 \rightarrow \text{RCOCH}_2\text{COCO}_2\text{H}$. Reissert (*Ber.* 30, 1030(1897)) showed that *o*- and *p*- $\text{O}_2\text{NC}_6\text{H}_4\text{Me}$ react thus: $\text{O}_2\text{NC}_6\text{H}_4\text{Me} + (\text{CO}_2\text{Et})_2 \rightarrow \text{O}_2\text{NC}_6\text{H}_4\text{CH}_2\text{COCO}_2\text{H}$. In the aliphatic compds. the negative groups (CO, CN, etc.) impart this mobility to the H atoms of Me; in the aromatic derivs. NO_2 imparts the same property to the Me group. v. Pechmann (*Ber.* 28, 855(1895)) found that the NO compd. of derivs. of MeNH_2 undergoes sapon. to give CH_2N_2 , thus: $\text{MeNHCOR} \rightarrow \text{MeN(NO)COR} \rightarrow \text{CH}_2\text{N}_2$. Similarly Noelting (*Ber.* 37, 2556(1904)) and Jacobson (*C. A.* 2, 1566) found that *o*- $\text{MeC}_6\text{H}_4\text{NHCOR}$ through the NO deriv. gives indazole which corresponds here to CH_2N_2 . The reactions $\text{O}_2\text{NOEt} + \text{PhNH}_2 \rightarrow \text{PhNHNNO}_2 + \text{EtOH}$ and $p\text{-O}_2\text{NC}_6\text{H}_4\text{OEt} + \text{PhNH}_2 \rightarrow \text{PhNHC}_6\text{H}_4\text{NO}_2 + \text{EtOH}$ are similarly analogous. The migration of O from N to C brings out this apparent close relation of the 2 groups in which the O atoms of NO_2 migrate to a C atom. These changes are very similar to the pinacolonic transformations (cf. *Atti accad. Lincei* 17, I, 311(1908)). Examples follow: $p\text{-MeC}_6\text{H}_4\text{NO}_2 \rightarrow p\text{-H}_2\text{NC}_6\text{H}_4\text{CO}_2\text{H}$; $o\text{-O}_2\text{NC}_6\text{H}_4\text{COH} \rightarrow o\text{-ONC}_6\text{H}_4\text{CO}_2\text{H}$; $o\text{-O}_2\text{NC}_6\text{H}_4\text{Me} \rightarrow o\text{-ONC}_6\text{H}_4\text{CH}_2\text{OH}$ (*Chem. Zentr.* 1908, II, 210); $o\text{-O}_2\text{NC}_6\text{H}_4\text{Me} \rightarrow p\text{-H}_2\text{NC}_6\text{H}_4\text{CO}_2\text{H}$ (*Z. angew. Chem.* 1900, 385). In the aliphatic series all the analogous transformations cannot be realized in 2 series. By oxidation the aliphatic NO_2 derivs. (*C. A.* 4, 2634) react thus: $2\text{RCH}_2\text{NO}_2 \rightarrow \text{O}_2\text{NRCH.CHRNO}_2$. With *o*- and *p*- $\text{MeC}_6\text{H}_4\text{NO}_2$ the compd. $\text{O}_2\text{NC}_6\text{H}_4\text{CH}_2\text{CH}_2\text{C}_6\text{H}_4\text{NO}_2$ was obtained. With MeNO_2 not only $\text{O}_2\text{NCH}_2\text{CH}_2\text{NO}_2$ but metazonic acid, $\text{O}_2\text{NCH}_2\text{CH:NOH}$, was obtained. Physiologically $\text{H}_2\text{NCO}_2\text{Et}$ has a hypnotic action while $p\text{-H}_2\text{NC}_6\text{H}_4\text{CO}_2\text{Et}$ is anesthetic. NH_2NH_2 is quite toxic but aliphatic diamines (putrescine, cadaverine) are physiologically inert. $p\text{-C}_6\text{H}_4(\text{NH}_2)_2$ is, however, toxic.

E. J. WITZEMANN

The chlorocarbonic esters. G. CAPPELLI. Rome. *Gazz. chim. ital.* 50, II, 8-12 (1920).—Me, Et and iso-Am chlorocarbonates are known. The importance of these compds. for organic synthesis makes a simple and convenient method of prepn. desirable. C. has worked especially with the Me and Et esters. The best results in the action of COCl_2 on MeOH or EtOH depends on always having the former present in excess. The use of solns. of COCl_2 in C_6H_6 , CCl_4 or in the chlorocarbonic esters only helped somewhat. Common wood charcoal used as an absorbent for COCl_2 gave the best results. The COCl_2 should be free from Cl and was best obtained from CCl_4 and SO_2 (oleum). Sometimes the prepn. of COCl_2 and the esters were carried out simultaneously. The alc. used contained 5 to 25% H_2O and gave optimum yields. The esters are best purified by allowing the excess COCl_2 to evap. spontaneously during a day. The app. used was set up as follows: The COCl_2 container is connected with a common wash bottle containing vaseline oil so that the velocity of gas flow may be observed. This is connected with a Woulfe bottle of 5 l. capacity having two tubulatures which serves as a safety receiver during absorption or to condense and hold back any CCl_4 . Next is the reaction chamber, a 5 l. Woulfe bottle with 3 openings through the 1st of which the gas enters, to the 2nd a graduated cylinder containing the alc. is attached and the 3rd is equipped with a reflux condenser which terminates in a good hood. For small preps. the connections are of glass, otherwise of lead tubes. Paraffined corks are best. 200 g. well dried charcoal are placed in the reaction chamber and this placed in a crushed ice bath. After cooling the COCl_2 is slowly admitted. After 20 mins. the flow is increased and the alc. is allowed to drop in rapidly. 1 kg. alc. may be esterified in 4 or 5 hrs. The crude ester after standing 20 hrs. is washed with H_2O and ice. The washed product is dried with CaCl_2 and fractionally distd. Yield 90%. E. J. WITZEMANN

Catalytic reactions. A. MAILHE. *Caoutchouc & gutta-percha* 17, 10584-5(1920).—In the presence of ThO_2 a mixture of PhOH and EtOH produce PhOEt. M. has reversed the reaction by heating ThO_2 to 460° , and passing over it steam and PhOEt. Two liquid layers are obtained, one containing water with a strong aldehyde reaction, and the other an oily layer containing the unchanged PhOEt, and part of the PhOH formed. In a similar manner PhNMe_2 has been broken up into PhNH_2 and MeOH. HCHO is always obtained as a product of the secondary reaction. J. B. T.

Mutarotation of dextrose in solutions of secondary sodium phosphate. HANS MURSCHHAUSER. *Biochem. Z.* 110, 181-92(1920); cf. *C. A.* 14, 3410.—An extensive tabular presentation is given with curves of the effect of various concns. of Na_2HPO_4 ranging from 0.001 *N* to *N* on the mutarotation of dextrose, demonstrating that the velocity const., practically is a linear function of the concn. of the soln. in Na_2HPO_4 , the velocity of mutarotation increasing with increasing phosphate concn.

F. S. HAMMETT

Chitin and chitin compounds in animals and plants. O. SCHMIEDEBERG. *Arch. exp. Path. Pharm.* 87, 76-85(1920).—Four compds. are described: (a) Hexaacetylchitin ($\text{C}_{42}\text{H}_{48}\text{N}_4\text{O}_{21}$); (b) pentaacetylchitin ($\text{C}_{34}\text{H}_{46}\text{N}_4\text{O}_{22}$); (c) tetraacetylchitin ($\text{C}_{26}\text{H}_{34}\text{N}_4\text{O}_{21}$); (d) triacetylchitin ($\text{C}_{18}\text{H}_{22}\text{N}_4\text{O}_{20}$). These forms can be transformed readily from one to another through the loss or addition of 1 part or more of Ac. A substance possessing the properties of hyaloidin can be prepd. from chitin. Compds. of the same compn. as the above, which were of animal origin, can be secured from fungi.

G. H. SMITH

The decomposition of glutamic acid and its salts by heat. VL. SKOLA. *Z. Zucker-ind. czechoslov. Rep.* 44, 347-51, 351-60, 363-8, 369-74(1920).—Glutamic acid, a constituent of certain sugar liquors, loses 1 mol. of water on heating, forming an anhydride, so-called *pyroglutamic acid*, which can be formulated as *tetrahydropyrrole- γ -carboxylic acid* or as a *lactam* of α -aminoglutaric acid. Complete dehydration occurs on heating the dry acid at 150 - 60° for 4 hrs. The reverse active *lactam acid* is obtained from an

active glutamic acid, *i. e.*, *d*-glutamic acid gives the *l*-lactam of α -aminoglutaric acid and *l*-glutamic acid changes to the *d*-lactam. At temps. above 185° racemization occurs not only of the glutamic acid but also of the pyroglutamic acid. At still higher temps. dry distn. takes place and pyrrole is produced among other substances. In H₂O the reaction is as follows: Glutamic acid \rightleftharpoons glutimic acid + water. The glutimic acid formed is identical with the lactam of glutamic acid. The equil. of this reaction is shifted in favor of the glutamic acid by the addition of a mineral acid. The addition of mineral bases causes the formation of salts of glutamic acid. On heating to higher temps. than 160° the active isomers become inactive. At temps. above 250° decompn. follows with the production of acetic, oxalic and succinic acids and other products. The NH₄ salt behaves differently than the corresponding alk. and alk. earth salts of the glutamic acid. The double NH₄ glutamate (?), when dried at 105° loses NH₃ and forms the single NH₄ salt. This change is complete at 115°. Subjecting this to a temp. of 160° for 5 hrs. causes the formation of *l*-pyroglutamic acid, which on heating to 185–190° produces an inactive compd. Boiling a soln. of NH₄ glutamate results in the evolution of NH₃ and on evapg. to dryness free glutamic acid is obtained. *Pyroglutamide*, by boiling with Ba(OH)₂ or through the action of HCl, finally decomp. into glutimic acid.

J. M. KENO

Transformation of naphthalene into anthracene and phenanthrene. G. SCHROETER. Berlin. *Brennstoff Chem.* 1, 39–41 (1920).—When tetrahydronaphthalene ("tetralin") (A) is digested at 30°–40° with 1% AlCl₃, about 30% of a mixt. of octahydroanthracene ("octhracene") (B), and octahydrophenanthrene ("octanthrene") (C), is formed. B and C are of interest because they can be reduced to anthracene and phenanthrene by passing over warm Cu, and the latter especially because of its close relation to the morphine alkaloids. Godchet has investigated at length (cf. C. A. 1, 1402, 3002–3; 2, 808) the constitution of an octahydroanthracene identical with B prepd. by Sabatier's reaction, and gives it the formula C₁₄H₁₆ $\begin{array}{c} \text{CH}_2 \\ \diagup \quad \diagdown \\ \text{C}_6\text{H}_8 \end{array}$ but S. disputes this conclusion on

the basis of the following synthesis: on warming A with ClCH₂COCl in the presence of P₂O₅, 1- and 2-tetralyl chlorides are formed; these with CH₃Na(CO₂Et)₂ give tetralacylmalonic esters, whose free acids, by loss of CO₂, give 1- and 2-tetroylpropionic acids. These may be reduced with Zn and HCl to 1- and 2-tetralylbutyric acids. The 2-acid is converted to its chloride by means of PCl₅, and this decomp. on gentle warming into a mixt. of 1-octhracene and 4-octanthrene which may be sepd. by the different solys. of their semicarbazones. The former was shown to be identical with Godchet's hexa-

hydroanthrene, thus proving that the structure of B is C₆H₈ $\begin{array}{c} \text{CH} \\ | \\ \text{C}_6\text{H}_8 \end{array}$ $\begin{array}{c} \text{CH} \\ | \\ \text{C}_6\text{H}_8 \end{array}$. The struc-

ture of C is likewise shown to be C₆H₈—CH $\begin{array}{c} \text{CH} \\ || \\ \text{C}_6\text{H}_8 \end{array}$ —CH

cipally to 1-octanthrene, which can be prepd. by warming 1-tetralinbutyryl chloride, and partly to 4-octanthrene, which is similarly formed from 2-tetralinbutyryl chloride. C, prepd. in pure form by hydrolysis of its sulfonic acid, f. 16.7°, b₁₀₆ 299°, b₁₁ 167.5°. The three secondary alcs. corresponding to 1-tetralone, 1-octhracene and 1-octanthrene, form substituted ureas on digestion with dil. solns. of urea. The action of AlCl₃ on A produces about 50% of a fraction b₁₂ 236°, which is a mixt. of β,β -ditetralyl (C₁₄H₁₄)₂ with higher phenylated compds. of B and C.

W. B. V.

The electrolysis of tetradecamethylenedicarboxylic acid. K. SROSIUS AND K. WIESLER. *Biochem. Z.* 108, 75–81 (1920).—Starting from castor oil as the base azelaic acid was prepd. by sapon. in alk. soln. Following Miller (*Ann.* 307, 384) di-Et azelate was formed and half sapond. in the calcd. amt. of alc. KOH soln. For

the electrolysis the app. of Brown and Walker (*Ann.* 261, 107(1891)) was used and the temp. kept low. 28 g. of the crude ester dissolved in 28 cc. H_2O were electrolyzed for 2.5 hrs. at a temp. of 30 to 40°, from which there was obtained 21% of the calcd. amt. of the di-Et tetradecamethylenedicarboxylate after fractional distn. *in vacuo*. After purification in EtOH the product was found to be a stable, cryst. white substance of fatty consistency. The ester was then sapond. by boiling alc. KOH for 2 hrs., the alc. removed by evapn., and the free acid obtained by the addition of dil. H_2SO_4 and extrn. with Et_2O . Further purification was obtained by extrn. with petr. ether, h. 25–40°. The final product m. 123°, was white, cryst., insol. in H_2O , sol. in petr. ether, EtOH, Et_2O , $CHCl_3$, and acetone. F. S. HAMMETT

Lignin and its reactions. P. KILASON. *Svensk Pappers-Tidning*, 23, 70(1920).—Previous work on lignin is reviewed. By examn. of the relation of ligninsulfonic acid to the salts of aromatic amines, principally β -naphthylamine, K. found that it is not a vanillin complex in the wood which occasions the lignin reaction hut coniferyl aldehyde. This explains how Wickelhaus could obtain decompn. products from woods which give lignin reactions. It was also found that coniferyl aldehyde and possibly also coniferyl alc. are essential constituents at least in lignin from pine wood. The investigation is to be continued. Cf. C. A. 14, 3229. W. SÄGERBLOM

The distillation of aqueous solutions of glycol monochlorohydrin. J. BANCERLIN AND G. RIVAT. *Bull. soc. chim* 25, 552–60(1919).—This is largely a continuation or an extension of the work of Razous and Gombert and in general confirms their results. Distn. was carried out by a Vigreux column with 8 condenser plates. The principal portion was re-fractionated, and yielded a few g. between 92.2 and 96° with a slight odor of aldehyde. The last fraction represented the max. proportion of monochlorohydrin; 42 to 58 of water. Tables and diagrams show m. and h. p. curves. There is apparently no justification of the conclusion of the presence of a definite hydrate for the mixt. at the minimum b. p. Attempts to conc. solns. by passing steam gave results inferior to those by fractional distn. L. G. SMITH

Organic molecular compounds. II. JAMES F. NORRIS AND DOROTHY M. TIBBETTS. *J. Am. Chem. Soc.* 42, 2085–92(1920); cf. C. A. 10, 1087.—With the hypothesis proposed in the first paper as guide a number of substances have been investigated as to their ability to form mol. compds. and the following new compds. have been discovered. *p*-Tetrabromotetraphenylethylene (A) compounds: Toluene, A.PhMe, from A dissolved in boiling C_6H_6 -free PhMe and allowed to cool, transparent needles slowly becoming opaque on standing; chlorobenzene, slowly effloresces in the air; ether, from a dil. soln. of A in Et_2O slowly evapd. in a current of air at -10° , transparent crystals becoming opaque in the air; *p*-xylene, very slowly effloresces in the air; methyl propyl ketone; ethyl propionate; propyl acetate. No compds. were isolated at room temp. with $CHCl_3$, EtOH, iso-AmOH, PhBr, *m*-xylene, PhI, petr. ether, AcOMe and HCO_2Et ; the fact that no compd. is formed with EtOH and with *m*-xylene can be utilized in sep. Me_2CO from the former and *p*-xylene from the latter. *p*-Tetrachlorotetraphenylethylene (B) compounds: Carbon tetrachloride, $B.2CCl_4$, loses its CCl_4 very slowly; acetone, $B.2Me_2CO$, formed in a $CaCl_2$ -ice bath, effloresces very rapidly at room temp.; benzene, $B.C_6H_6$; methyl ethyl ketone, $B.MeCOEt$; AcOEt gives no compd. *p*- ClC_6H_4CPh : CPH_3 (C) forms at -15° a carbon tetrachloride compound, $C.2CCl_4$, which rapidly effloresces at room temp.; no compds. were isolated with C_6H_6 , $MeCOPr$ or Me_2CO . A compound $PhC_6H_4CH(OH)Ph.CCl_4$ is obtained at -10° hut Me_2CO gives no addition product. A very unstable compound $2PhC_6H_4CHPhCl.CCl_4$ is formed at -15° ; here again Me_2CO does not yield an addition product. CHAS. A. ROUILLER

Reaction between alcohols and aqueous solutions of hydrochloric and hydrobromic acids. II. JAMES F. NORRIS AND ROBERT S. MULLIKEN. *J. Am. Chem. Soc.* 42, 2093–8(1920); cf. C. A. 10, 1646.—The behavior towards aq. HBr and HCl of

certain polyatomic alcs. and of a few monoatomic alcs. with negative substituents or belonging to groups not previously investigated has been studied. All reacted with HBr, strengthening the conclusion earlier drawn that HBr may be used to test for alc. OH groups. Aq. HCl replaced the HO group in certain alcs. containing negative substituents, but its failure to react in other cases shows clearly that other influences than those of negativity serve to activate the HO groups. $(\text{CH}_2\text{OH})_2$ heated with HBr (d. 1.49) gave a mixt. of the bromohydrin and $(\text{CH}_2\text{Br})_2$, the proportions of which varied with the conditions. With propylene glycol, profound changes with formation of deeply colored oils, resulted. Trimethylene glycol gave 82% bromide and some bromohydrin. Glycerol yielded 46% dibromo- and 13% monobromohydrin. $(\text{CH}_2\text{OH})_2$ heated with a large excess of HCl (d. 1.17) gives 12% chlorohydrin, but $\text{BrCH}_2\text{CH}_2\text{OH}$ does not react with HCl. Propylene glycol undergoes a deep-seated change when heated with HCl but gives 5% chlorohydrin; trimethylene glycol gives a trace of chloride and 28% of chlorohydrin. The latter is converted to a small extent into the former when boiled with HCl; the bromohydrin behaves similarly. Glycerol forms a trace of di- and about 9% of monochlorohydrin; pinacol hydrate slowly gives the chloride at room temp.; cyclohexanol yields 93% of the chloride, menthol 41%, borneol 30%, cumyl alc. practically 100%.

CHAS. A. ROUILLER

Preparation of dichloroacetic acid from chloral. GEORGE W. PUCHER. *J. Am. Chem. Soc.* 42, 2251-9 (1920).—The results of this work confirm Kötze's views (*C. A.* 5, 886) as to the mechanism of the reaction. The $\text{Cl}_2\text{CHCO}_2\text{H}$, b_p 102°, can be obtained in 60-5% yield by treating 1 kg. $\text{CCl}_3\text{CHO} \cdot \text{H}_2\text{O}$ in 3 l. H_2O at 40°, with stirring, with 400 g. com. NaCN in 1100 cc. H_2O at such a rate that the temp. does not rise above 50°, keeping 1 hr. longer at 50°, concg. on the H_2O bath to a pasty mass, suspending in 2.5 l. C_6H_6 in ice, passing in HCl gas for 4-5 hrs., filtering, drying with Na_2SO_4 and removing the solvent by distg. from a bath at 160°. Corrosion tests with Fe, Cu, Al and Pb showed that at 20° none of these metals is readily attacked by the acid but the corrosion increases with diln. and rise in temp. Al appears to be the most resistant to the action of the pure acid at a high temp. and Pb and Cu to that of the dil. acid. *Methyl diethoxyacetate*, obtained in 44% yield from 50 g. $\text{Cl}_2\text{CHCO}_2\text{H}$ slowly added to 30 g. Na in EtOH, heated 2 hrs. on the H_2O bath, cooled, filtered from the NaCl, freed from the excess of alc. *in vacuo* and heated 3 hrs. on the H_2O bath with 75 g. Me_2SO_4 , b. 184-5°, does not solidify -81°, d_{20} 1.0049.

C. A. ROUILLER

Possible asymmetry of the aliphatic diazo compounds. C. S. MARVEL with W. A. NOYES. *J. Am. Chem. Soc.* 42, 2259-78 (1920).—As in the prepn. of aliphatic diazo compds. one of the N atoms comes from NH_3 and the other from HNO_3 , the electronic formula for such compds. would be $\text{R}-\text{C}-\text{R}'$. Such a mol. would be asym.



and optical isomers would be expected to exist. An effort has been made to prep. such isomers by treating active NH_2 esters with HNO_3 but so far all such attempts have failed either because (1) the two N atoms are alike; (2) they are different but the compds. racemize during the reactions; or (3) the Curtius formula does not correctly represent the structure of these compds. $\text{PhCH}(\text{NH}_2)\text{CO}_2\text{H}$ (A) is obtained in 105-10 g. yield by treating 100 g. NaCN in 400 cc. H_2O and 106 g. NH_4Cl with 212 g. BzH in 400 cc. MeOH, shaking thoroughly, letting stand 1-1.5 hrs., pptg. with 1 l. H_2O , taking up the oily product in 1 l. C_6H_6 , extg. from the C_6H_6 twice with 600 cc. of HCl (1:1), refluxing 2 hrs. to hydrolyze the nitrile and pptg. with NH_4OH ; 200 g. of the acid in 1 l. alc. and 70-80 g. HCl refluxed 3 hrs. gives 142-153 g. of the *dl*-ester (B), b. 114-5°, n_D^{20} 1.500, quant. gives with HCl in C_6H_6 the hydrochloride, m. 200°. *dl*-Ethyl acetylaminophenylacetate (11 g. from 11.5 g. B allowed to stand 2 hrs. with 10 cc. Ac_2O and then heated 1

hr. on the H_2O bath), crystals from ligroin, m. $65-6^\circ$. *d*-Ethyl carbethoxyaminophenylacetate (7 g. from 18 g. B in 50 cc. Et_2O slowly treated with 5.5 g. $ClCO_2Et$ and allowed to stand 1 hr.), crystals from ligroin, m. 57° . Neither of the above 2 compds. yielded NO derivs. with nitrous oxides in Et_2O , $NaNO_2$ in glacial AcOH or $NaNO_2$ in HCl. Impure N_2CPhCO_2Et can be obtained in fairly good yields by the method described below for N_2CBuCO_2Et but on attempting to dist. the crude product it decomps. with formation of $PhCH(OH)CO_2Et$. By means of 232 g. *d*-camphorsulfonic acid 151 g. of A can be resolved (cf. Betti and Mayer, *C. A.* 2, 2800) into 107-14 g. of the *l*-salt, $[\alpha] -43.5^\circ$ to -44° (206 g. of which gives 80 g. pure *l*-A), and 65-70 g. *d*-A, $[\alpha] 127-46^\circ$. *l*-B-HCl (83 g. from 81 g. *l*-A), $[\alpha] -84.6^\circ$. *l*-Ethyl acetylaminophenylacetate (about 1 g. from 4.3 g. *l*-B-HCl in 10 cc. H_2O treated with NH_4OH , extd. with C_2H_5 , and refluxed 1-2 hrs. with 2.5 g. Ac_2O), crystals from ligroin, m. $69-70^\circ$, $[\alpha] -138.7^\circ$ (alc.). Diazotization of 21.5 g. *l*-B-HCl in NaOAc with $NaNO_2$ -AcOH gave 0.0087 g. of a substance which, made up to 20 cc. in Et_2O , showed a rotation of -0.1° in a 2-dm. tube while from 10 g. of the *l*-B-HCl and 8 g. $AgNO_3$ was obtained a product which in 25 cc. Et_2O showed a rotation of -4.21° in a 2-dm. tube; titration with I showed that this Et_2O soln. contained 0.165 g. diazo ester; 15 cc. of the soln. shaken with 20% H_2SO_4 until the yellow color was destroyed and again made up to 15 cc. showed a rotation of -0.96° in a 2-dm. tube. The expt. may indicate the existence of an active diazo ester hydrolyzed to an active HO ester; it was shown exptly., however, that 20% H_2SO_4 racemizes active $PhCH(OH)CO_2Et$, so that it seems probable that the rotation observed in the diazo ester soln. was due to the presence of *l*- $PhCH(OH)CO_2Et$. In attempting to prep. the latter (Fischer and Weichbold, *C. A.* 2, 2237), 21.5 g. *l*-B-HCl in 130 cc. *N* H_2SO_4 at 0° treated with 6.4 g. $NaNO_2$ in 10 cc. H_2O , kept 1 hr. at 0° and gradually allowed to warm up to room temp. gave 7.5 g. $PhCH(OH)CO_2Et$, $b_D^{15} 130-5^\circ$, m. about 30° , with a rotation of -5.25° in a 1-dm. tube, thus confirming the previous observation that most of the ester is racemized. The nitrite of *l*-B (from $AgNO_3$ and *l*-B-HCl in Et_2O) allowed to stand 2 days in Et_2O with Ac_2O gives *l*- $PhCH(NHAc)CO_2Et$, m. $68-70^\circ$. Methylphthalimidine (C), $b_D^{10} 180^\circ$, m. 100° , was obtained in 25 g. yield from 80 g. of the phthalazone by Gabriel's method (*Ber.* 26, 706(1893)); 20 g. in 200 cc. HCl (1:1) at 0° with 10 g. $NaNO_2$ in 20 cc. H_2O gives 20 g. nitrosomethylphthalimidine, crystals from 50% alc., m. $86.5-7.0^\circ$; attempts to convert this into *o*- $EtO_2CC_6H_4CMeN_2$ by treating 3.5 g. of it in 300 cc. Et_2O at -10° with 1 g. Na in 10 cc. alc. and, after about 0.5 hr., with CO_2 , gave only a small amt. of a reddish oil changing to a yellow solid, m. 220° . All efforts to resolve C failed. Ethyl α -diazocaproate (13 g. from 50 g. $ClH_2NCH_2BuCO_2Et$ in 150 cc. H_2O at -10° treated with 60 g. NaOAc and 60 g. $NaNO_2$ and, in the course of 1 hr., with 75 cc. AcOH, kept 3.5 hrs. at -5° to -10° , washed with H_2O and $NaHCO_3$, allowed to stand over solid $Ba(OH)_2$, dried with $CaCl_2$ and distd. *in vacuo*), $b_D^{10} 75-8^\circ$, is lemon-yellow, lighter than H_2O , $n_D^{25} 1.453$, solidifies in CO_2 - Et_2O ; steam distn. destroys about $2/3$ of it and in no way purifies it; 10 g. refluxed 1 hr. with 50 cc. of 10% AcOH gives about equal parts of $BuCH(OH)CO_2Et$ and $PrCH:CHCO_2Et$. *l*-Ethyl α -aminocaproate (11.5 g. from 13 g. of the acid), $b_D^{12} 86-7^\circ$, $\alpha -11.65^\circ$ in a 1-dm. tube; hydrochloride (14 g.), $[\alpha]_D -7.25^\circ$. *d*-Ester (11 g. from 13 g. of the acid), $b_D^{10} 85^\circ$, $\alpha 6.15^\circ$ in a 1-dm. tube; hydrochloride (13 g.). Attempts to prep. active diazo compds. from these esters failed. Diazotization of 13 g. of the *d*-ester hydrochloride in *N* H_2SO_4 gave 4 g. product $b_D^{10} 65-70^\circ$ and $87-90^\circ$, consisting of apparently about equal amts. of HO ester and unsatd. ester and which with 2 g. Et_2O showed a rotation of 0.7° in a 1-dm. tube. α -Bromocaproyl chloride (37 g. from 50 g. of the acid and 28 g. $SOCl_2$), $b_D^{10} 102-5^\circ$; 65 g. of this and 350 cc. *N* NaOH added in alternate portions to 22.5 g. glyocoll in 300 cc. *N* NaOH at 0° gave 58 g. α -Bromocaprolylglycine, crystals pptd. from Et_2O by petr. ether, m. $114-5^\circ$; 30 g. allowed to stand 4 days in 150 cc. NH_4OH (d. 0.9) yields 19 g. α -aminocaprolylglycine, m. 226° ; 10 g. in 100 cc. alc. satd.

with HCl and refluxed 15 min. yields 14.5 g. of the non-crystallizable *ethyl ester hydrochloride*, 14 g. of which in 50 cc. of H₂O with 10 g. NaOAc, 5 g. NaNO₂ and 6 cc. AcOH yields 3-4 g. *ethyl α-hydroxycaproylglycine*, crystals from Et₂O-petr. ether, m. 90-1°. *α-Aminocaprylic acid* is obtained in 70-5 g. yield from 55 g. NaCN in 100 cc. H₂O and 57 g. NH₄Cl treated with 114 g. heptaldehyde in 100 cc. MeOH, allowed to stand overnight, extd. with Et₂O, evapd. and refluxed 2.5 hrs. with 350 cc. HCl and 150 cc. H₂O; 50 g. yields 40 g. of the *ethyl ester*, b₁₀ 110°, n_D^{20} 1.436, whose *hydrochloride* m. 76-7°. Attempts to obtain a diazo compd. gave under the best conditions (from 18.7 of the NH₂ ester in 50 cc. H₂O, 6 cc. AcOH, 20 g. NaOAc and 15 g. NaNO₂ covered with Et₂O, slowly treated with 20 cc. AcOH and allowed to stand 4 hrs. at 0°) 9 g. of a product b₁₀ 105-10°, with 8.15% N (calcd. 14.14). EtMeC:NOH (203 g. from 250 g. NH₂OH·H₂SO₄ in 1500 cc. H₂O allowed to stand 24 hrs. with 216 g. EtCOMe and 160 g. Na₂CO₃), b. 150-5°; 90 g. in 1 l. alc. treated in the course of 1 hr. with 150 g. Na and boiled with occasional addition of more alc. (about 300 cc. in all) until all the Na was dissolved gave 40 g. EtMeCHNH₂, b. 66-70°, 40 g. of which in 100 cc. Et₂O with 30 g. ClCO₂Et in Et₂O gave 30 g. EtMeCNHCO₂Et, b₁₄ 87-8°; all attempts to prep. a NO deriv. of the latter failed.

CHAS. A. ROUILLER

Syntheses in the cinchona series. VI. Aminoazo and hydroxyazo dyes derived from certain 5-amino cinchona alkaloids and their quinoline analogs. WALTER A. JACOBS AND MICHAEL HEINELBERGER. *J. Am. Chem. Soc.* 42, 2278-86 (1920); cf. *C. A.* 14, 2797.—A number of aminoazo dyes (with the N:N group probably in the 8-position of the quinoline portion of the mol.) have been prepd. by coupling 5-aminodihydroquinine (A), 5-aminodihydroquinidine (B) and 5-aminoethyldihydrocupreine (C) with diazo compds. in dil. AcOH. These substances are usually red, well crystd. compds. sol. in neutral solvents with orange-red and in dil. mineral acids with deep purple color. If the solus. in mineral acids are boiled the color changes very rapidly from purplish red to brown-red with replacement of the 5-NH₂ group by HO; 5-15 min. boiling on the H₂O bath with a mixt. of equal parts of 1:1 HCl and alc. suffices for this replacement to be quant. This lability of the NH₂ group is not dependent on the influence of the cinchona mol. as a whole for it is also found in the dyes derived from 5-amino-6-methoxyquinoline; a MeO or EtO group in position 6 apparently contributes markedly to the lability of the 5-NH₂ group. These hydroxyazo dyes also cryst. well and in spite of the HO group are insol. in dil. alkali, but after making an alc. soln. alk. it may be dild. freely without pptg. the dye, which can, however, be pptd. by NH₄Cl. *5-Amino-8-phenylazodihydroquinine* (2.2 g. from 1 g. PhNH₂ in 30 cc. N HCl diazotized with 10 cc. N NaNO₂ added slowly to 3.4 g. A in 20 cc. N AcOH, 180 cc. H₂O and 20 cc. of satd. NaOAc at about 10°), red needles with approx. 1 H₂O from 95% alc., m. 155-7°, sol. in concd. H₂SO₄ with intense purple color; *hydrochloride*, deep brown micro-needles. *5-Hydroxy-8-phenylazodihydroquinine* (4.2 g. from 5 g. of the NH₂ dye boiled 15 min. with 25 cc. alc. and 25 cc. of 1:1 HCl), orange-brown platelets with 1 H₂O, effervesces 145-8°, sol. in dil. H₂SO₄ with orange-red and in concd. acid with cherry-red color, forms an insol. gelatinous salt in dil. HCl. *5-Amino-8-p-sulfo-phenylazodihydroquinine* (4 g. from 3.4 g. A), orange needles with about 0.5 H₂O from 50% alc., darkens above 180°, decomps. about 245°, yields in not too dil. HCl or H₂SO₄ dark, cryst., difficultly sol. salts, dissolves in concd. H₂SO₄ with bright red color, quickly reduced in 50% AcOH by SnCl₂ to 5,8-diaminodihydroquinine. *5-Hydroxy compound* (1.8 g. from 2 g. of the NH₂ dye), scarlet needles with approx. 1 H₂O from 50% alc., decomps. 275°, forms brown-orange salts with dil. HCl and H₂SO₄, dissolves in concd. H₂SO₄ with deep scarlet color. *5-Amino-8-p-methoxyphenylazodihydroquinine* (2.8 g. from 1.23 g. *p*-MeOC₆H₄NH₂ in 15 cc. 2 N HCl and 2 cc. of 5 N NaNO₂ treated at 0° with 3.4 g. A in 10 cc. of N AcOH and enough satd. NaOAc to bind the free HCl), red microneedles and platelets from 95% alc., m. 150-3°, sol. in concd. mineral acids with deep violet-

blue color changing to brownish red on diln., in concd. H_2SO_4 with deep red color. *8-p-Ethoxy homolog* (2.6 g. from 1.75 g. $p\text{-EtOC}_6\text{H}_4\text{NH}_2\cdot\text{HCl}$), long thin narrow orange platelets (apparently solvated) from CHCl_3 -ligroin, decomp. $202\text{--}3^\circ$, sol. in mineral acids with blue-violet color changing to dark wine-red on diln., with deep purple color in concd. H_2SO_4 . *5-Amino-8-phenylazodihydroquinidine*, orange-red leaflets with 1.5 H_2O from 85% alc., m. 135° , turns dark brown when dehydrated and then m. about $140\text{--}5^\circ$, sol. in concd. H_2SO_4 with intense violet color; *hydrochloride*, brown hair-like needles with greenish luster. *5-Amino-8-phenylazoethylidihydrocupreime* (3 g. from 3.55 g. C), red needles with 1 H_2O from 95% alc., sinters 145° , m. $150\text{--}5^\circ$, sol. in dil acids and in concd. H_2SO_4 with purple color. *5-Hydroxy compound*, red needles with 3.5. H_2O from 85% alc., sinters 85° , m. 90.5° , sol. in 10% H_2SO_4 with deep brown-red and in concd. acid with deep purple color, softens (anhydrous) above 100° , sinters $110\text{--}20^\circ$, m. about 200° (decompn.). *5-Amino-8-phenylazoquinoline*, garnet leaflets from 50% alc., m. $209\text{--}11^\circ$, sol. in dil. acids and concd. H_2SO_4 with deep red color; boiled with 100 parts of 10% HCl (about 1 hr. is required for complete reaction) it changes into the *5-hydroxy compound*, slightly purplish red needles from alc., m. $164\text{--}5^\circ$, sol. in concd. H_2SO_4 with scarlet color becoming orange on diln. *5-Amino-6-methoxyquinoline*, obtained in 90% yield by reduction of the NO_2 compd. with SnCl_2 , yellow platelets and needles from ligroin, m. $154\text{--}6^\circ$, sol. in acids with reddish orange color. *5-Amino-6-methoxy-8-phenylazoquinoline*, thin deep red rods with green reflex from 85% alc., m. $163\text{--}4^\circ$, sol. in dil. acids with purplish red, in concd. H_2SO_4 with purple color, converted by 30 parts of a mixt. of equal vols. of 1:1 HCl and alc. on the H_2O bath into the *5-hydroxy compound*, broad scarlet needles from alc., m. $181\text{--}3^\circ$, sol. in boiling dil. mineral acids with deep orange-red, in concd. H_2SO_4 with wine-red color.

CHAS. A. ROULLER

Condensation of formaldehyde with *o*-nitrophenol. JACOB B. FISHMAN. *J. Am. Chem. Soc.* 42, 2288-97(1920).—In view of the pharmacological properties (local anesthetic action) of PhCH_2OH and of saligenin and its ethers and homologs, theoretically a combination of the greatest interest would be $3,4\text{-(HO)}_2\text{C}_6\text{H}_3\text{CH}_2\text{OH}$ on account of its close relationship to adrenaline; the constitution of such a compd. would be favorable for both anesthetic and constrictor action, *i. e.*, the substance might be expected to exhibit the combined physiol. effects of PhCH_2OH and catechol. The purpose of the present work was to develop the synthetical aspects of the subject. The $o\text{-O}_2\text{NC}_6\text{H}_4\text{OH}$ (A) was obtained in 51% yield by direct nitration of PhOH (Hart, *C. A.* 4, 2945). From a careful study of the condensation of HCHO with A (Stoermer and Behn, *Ber.* 34, 2459(1901)), it is concluded that (1) a high concn. of HCl is essential for max. condensation; (2) to avoid side reactions and to obtain the max. yield of $4,3\text{-HO(O}_2\text{N)-C}_6\text{H}_3\text{CH}_2\text{OH}$ (B) it is not advisable to use more than the calcd. amt. of HCHO ; (3) working with mol. proportions of HCHO it is not advisable to continue the heating more than 6 hrs. to obtain a max. yield of B. From 200 g. HCHO (40%), 300 g. A and 1500 g. concd. HCl digested 6 hrs. were obtained 144 g. unchanged A and 63 g. of a cryst. mixt. of B and its isomer C (see below); the remainder of the product is an amorphous substance insol. in hot H_2O and which does not dist. with steam. B and C are best sepd. by digestion in EtOH with alc. KOH , S. and B.'s compd., B, m. 97° , dissolving as the K salt, while C forms an insol. K salt. C seps. from H_2O in yellow needles, m. 75° , and is *3-nitro-2-hydroxybenzyl alcohol*, easily sol. in cold concd. H_2SO_4 with deep red color; its K salt is bright red. The relative amts. of B and C formed are about 3:1. KMnO_4 oxidizes C to $2,3\text{-HO(O}_2\text{N)C}_6\text{H}_3\text{CO}_2\text{H}$, m. 144° . With MeOH , NaOH and MeI C gives *3-nitro-2-methoxybenzyl alcohol*, needles from petr. ether- Et_2O , m. 42° (yield, about 60%), oxidized by alk. KMnO_4 to *3-nitro-2-methoxybenzoic acid*, needles from alc., m. $191\text{--}2^\circ$ (Keller, *C. A.* 2, 2086, gives 110° for the acid prepd. by alkylating $2,3\text{-HO(O}_2\text{N)C}_6\text{H}_3\text{CO}_2\text{H}$).

CHAS. A. ROULLER

Some derivatives of 3-nitro-4-hydroxybenzyl alcohol. JACOB B. FISHMAN. *J. Am. Chem. Soc.* 42, 2297-302(1920); cf. preceding abstr.—4,3-HO(O₂N)C₆H₃CHO, needles from H₂O, m. 142°, is easily obtained from 5 g. of the alc. (A) and 1 mol. KOH in 100 cc. H₂O treated slowly at 90-100° with 3 g. KMnO₄. With excess of KMnO₄ A gives 4,3-HO(O₂N)C₆H₃CO₂H, m. 183-4°. Attempts to reduce A with Zn dust and alc., SnCl₂ and HCl and FeSO₄ and NH₄OH gave no definite cryst. product. 4,3-MeO-(O₂N)C₆H₃CH₂OH, needles from H₂O, m. 69°, is obtained in about 70% yield from A, KOH and MeI in MeOH, and the EtO compd., needles from H₂O, m. 66°, in 60% yield. 3-Amino-4-methoxybenzyl alcohol (B), obtained in 75% yield from the NO₂ compd. with NH₄OH-FeSO₄, crystals from C₆H₆, m. 95°. 4-Ethoxy homolog, flat prisms, m. 83°. From 5 g. B in 12 cc. concd. HCl and 75 cc. H₂O slowly treated below 10° with 8 g. NaNO₂ in 40 cc. H₂O, allowed to stand 1 hr., warmed to 60° for 20 min., evapd. to dryness at 60° *in vacuo* and extd. with hot abs. alc. is obtained the very stable 2-methoxy-5-hydroxymethylbenzenediazonium chloride, cream-colored plates, decomp. 131° without melting and with evolution of red fumes, forms an aq. soln. neutral to litmus even after boiling, decompd. by concd. H₂SO₄ with evolution of N. 2-Ethoxy homolog, decomp. 131°. 3-Cyano-4-methoxybenzyl alcohol, from the diazo compd. by the Sandmeyer methods, needles from H₂O m. 81°; yield, about 50%. CHAS. A. ROUILLER

Action of ammonia and amines on the substituted ureas and urethans. II. Allophanic ester. F. B. DAINS AND E. WERTHEIM. *J. Am. Chem. Soc.* 42, 2303-9 (1920); cf. C. A. 13, 2038.—It has been found that H₂NCONHCO₂Et (A) reacts with primary amines, RNH₂, to form both RNHCONHCO₂Et + NH₃ and RNHCONHCO₂NH₂ + EtOH; at higher temps. mono- and disubstituted ureas are formed, probably because of the continued action of the amines on the biuret, RNHCONHCONHR. A, m. 189-92°, is obtained in 62-5% yield from 1 mol. ClCO₂Et and 2.1 mols. urea heated 2-3 hrs. on the H₂O bath under a reflux, treated with H₂O while still warm and filtered with suction. Heated with slightly more than 1 mol. PhNH₂ 1 hr. at 120-5° it gives PhNHCONHCO₂Et, m. 105°, while with 2 mols. PhNH₂ 1 hr. at 125° it forms in addition diphenylbiuret, silky needles from alc., m. 209-10°, and at 160-70° it gives both mono- and diphenylurea. With *o*-anisidine 2 hrs. at 130° it yields ethyl *o*-anisylallophanate, needles from alc., m. 125°, sol. in dil. NaOH, also obtained from MeOC₆H₄NHCONH₂ and ClCO₂Et; with 2 mols. amine the main product is di-*o*-anisylbiuret, needles from Me₂CO or alc., m. 211-2° (partial sublimation); at 175° only di-*o*-anisylurea, m. 182°, is formed. With 1 mol. *o*-toluidine 4 hrs. at 115-6° is formed *o*-MeC₆H₄NHCONHCO₂Et, m. 133°; with 2 mols. amine at 160-90° are obtained mono- and di-*o*-tolylurea, m. 189° and 245-7°, resp., and di-*o*-tolylbiuret, needles from alc., m. 204-5°. With *m*-toluidine no MeC₆H₄NHCONHCO₂Et could be obtained; the products were mono- and di-*m*-tolylurea, m. 143° and 217°, resp., and di-*m*-tolylbiuret, needles from alc., m. 179°. *m*-O₂NC₆H₄NH₂ in kerosene 2 hrs. at 125° gave *m*-O₂NC₆H₄NHCONHCO₂Et, m. 189-90°; at 145° was formed di-*m*-nitrophenylbiuret, yellow crystals from alc., m. 215-6°; at higher temps. *m*-O₂NC₆H₄NHCONH₂, m. 165°, and di-*m*-nitrophenylurea, m. 133-4°. *p*-BrC₆H₄NH₂ does not seem to react below 120°; after 2 hrs. at 120° is obtained di-*p*-bromodiphenylbiuret, pearly plates from PhNO₂, decomp. 280°; at 140° *p*-bromophenylurea, plates, decomp. above 270°; at 150° di-*p*-bromophenylurea. H₂NCONHCO₂Me (B) is obtained in 75% yield from ClCO₂Me and 2 mols. urea. PhNHCONHCO₂Me could under no conditions be obtained from B and PhNH₂. With 1 mol. PhNH₂ 2 hrs. at 150° is formed PhNHCONHCONHPh, scales from alc., Me₂CO or H₂O, usually m. 164-7° but after further crystn. and drying at 100° they m. and darken 155°. At 150-70° diphenylbiuret is formed; above 170° chiefly CO(NHPh)₂; H₂NCONHPh was isolated only in 1 case. *o*-Toluidine gave *o*-tolylbiuret, m. and sublimed 179-80°, in various amts. at 140-70°; other products isolated were di-*o*-tolylbiuret and mono- and di-*o*-tolylurea; no tolylallophanate was

formed. $\text{BzNHCONHCO}_2\text{Et}$ and PhNH_2 at 150° gave BzNHCONHPh , m. 209° . At 140° A and PhNHNH_2 give *1-phenyl-4-carbethoxysemicarbaside*, $\text{PhNHNHCONHCO}_2\text{Et}$, scales from alc., m. $175-6^\circ$, losing alc. above its m. p. with the formation of 1-phenylurazole (C), m. $263-4^\circ$ (which is also produced quant. when A and PhNHNH_2 are heated above 140°), while with NH_3 it gives a compd. m. 195° . With B and PhNHNH_2 the intermediate carbomethoxy compd. could not be isolated; after a short time at 150° there was no reaction but on longer heating at this temp. or a few min. at 153° only C was formed.

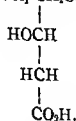
CHAS. A. ROUVILLER

Methodides of the condensation products of some cyclic aldehydes with quinaldine and α -picoline, and their possible value as indicators in acidimetry. LOUIS F. WERNER. *J. Am. Chem. Soc.* 42, 2309-14(1920).—2-[3-Methoxy-4-hydroxystyryl]quinoline methiodide (A) in H_2O with NaOH gives a deep fuchsin-colored liquid instead of the expected ppt. of the quaternary hydroxide. A study of the methodides of the condensation products of various cyclic aldehydes with quinaldine (B) showed that in order to show these properties of an indicator there must be a phenolic HO group in the nucleus of the aldehyde used in the condensation. Preliminary expts. on the possibility of using these compds. as indicators indicate that they may be so used if the HI formed by their hydrolysis in H_2O is first neutralized with alkali. 2-[β -Furylvinyl]quinoline, crystals from ligroin, m. 56° , is prepd. by heating 9 g. B, 6 g. furfural and 2 g. ZnCl_2 , 5 hrs. on the H_2O bath; heated 4-5 hrs. with excess of MeI in the dark at 140° it yields the methiodide, orange needles with steel-blue reflex from H_2O , m. $226-2^\circ$, forms a lemon-yellow neutral or acid and a colorless alk. soln. but the change in color is so slight that the substance is of no value as an indicator. 2-[3,4-Methylenedioxy-styryl]pyridine methiodide, from the pyridine heated 4 hrs. in the dark at 100° with excess of MeI, lemon-yellow microcryst. powder from H_2O , m. 276° , forms a yellow neutral or acid and a colorless alk. soln. 2-Styrylquinoline methiodide, bright orange acicular crystals from H_2O , m. 235.2° (decompn.), forms faintly yellow neutral or acid and colorless alk. solns. 2-[2-Hydroxystyryl]quinoline methiodide, bright orange cryst. powder from H_2O , m. 236° ; 1 cc. of a 0.1% soln. in 250 cc. H_2O is turned blood-red by 1 drop of 0.1 N NaOH but changes to a pale lemon in a few sec., the blood-red color becoming permanent only after 0.4 cc. of alkali has been added; this color is discharged by 0.12 cc. of 0.1 N HCl but restored by 0.1 cc. of 0.1 N NaOH. 2-[4-Hydroxystyryl]quinoline methiodide, bright orange cryst. powder from H_2O , m. 250.8° (decompn.), behaves like the preceding compd. towards acids and alkalis. A is a brick-red cryst. powder, m. 267° (decompn.), behaves like the preceding 2 compds. towards acids and alkalis, being fuchsin-red in acid and lemon-yellow in neutral soln. 2-[3,4-Dihydroxystyryl]quinoline m. 244.6° ; methiodide, brownish orange powder from H_2O , m. 266.8° (decompn.); 1 cc. of a 0.15% soln. in 250 cc. H_2O is turned permanently grape-colored by 0.4 cc. of 0.1 N NaOH and lemon-yellow by 0.1 cc. of 0.1 N HCl.

CHAS. A. ROUVILLER

The C₇-saccharinic acids. I. Resolution of *dl*-2,3-dihydroxybutyric acid into the optically active components. The derivatives of these acids. J. W. E. GLATTFELD AND GEORGE E. MILLER. *J. Am. Chem. Soc.* 42, 2314-21(1920); cf. C. A. 12, 1777. —Owing to the lack of data on the properties and consts. of the active C₇-saccharinic acids their prepn. in a systematic manner has been undertaken; the present paper is a report on the prepn. of the first pair, the 2,3-dihydroxybutyric acids (A). The *dl*-A was prepd. by Hanriot's method (cf. Nef, C. A. 5, 85); the phenylhydrazide m. $100-1^\circ$ (N. gives 99°); the constitution of the acid was confirmed by its prepn. from $\text{CH}_2\text{:CHCH}_2\text{CO}_2\text{H}$ with KMnO_4 . By fractional crystn. from ahs. alc. its hucrine salt was sep'd. into a fraction with $[\alpha]_D^{20} -29.42^\circ$ (4% aq. soln.), the free acid from which, a clear yellow mobile oil, showed, in about 4% aq. soln., $[\alpha]_D -8.29^\circ$; titration showed that in such solns. the acid was present to the extent of about 14.4% as free acid, the rest as

lactone. The gummy barium salt (which became cryst. on triturating with abs. alc.), vacuum-dried, showed $[\alpha]_D^{20}$ 1.48° (0.4 g. in 9.6 g. H_2O); the calcium salt, 2.47°; phenylhydrazide, crystals from AcOEt, m. 102–3°, $[\alpha]$ 1.71° (0.54 g. in 12.06 g. H_2O). The free acid from the non-crystallizable brucine salt, a light yellow mobile oil purified through the Ba salt, showed $[\alpha]$ 8.00° (0.83 g. in 19.92 g. H_2O); barium salt, $[\alpha]$ –1.48° (1 g. in 24 g. H_2O); phenylhydrazide, gum very sol. in AcOEt. The acid with $[\alpha]$ –8.29°, heated 2 hrs. at 65° with 10 parts of HNO_3 (d. 1.305), is converted into natural *l*-malic acid and is therefore *l*-A, CH_2OH



CHAS. A. ROULLER

Preparation of optically active hydrazines. I. Preparation of *dl*-*p*-trimethylethylphenylhydrazine. Isolation of pure *d*-*p*-trimethylethylaniline. J. W. E. GLAZIER AND C. H. MILLIGAN. *J. Am. Chem. Soc.* 42, 2322–8 (1920).—Hoping to obtain some fairly easily prepd. reagent which might be used to supplement the alkaloids in the resolution of the saccharinic acids (see preceding abstr.), G. and M. undertook to prep. optically active hydrazines. The present paper is a report of their first efforts in this direction. Although they have prepd. *dl*-trimethylethylphenylhydrazine (A), they have failed to resolve it into its active components, probably on account of the instability of the free hydrazine in the air under ordinary conditions. Some of its derivs. are perfectly stable compds., however, and after much experimentation, one of the active derivs., *d*-*p*-trimethylethylaniline (B), was obtained pure. G. and M. have not yet had time to convert this into the corresponding hydrazine by diazotization and reduction. Trimethylphenylethylene, $Me_3C:CMePh$, h. 187–8°, is prepd. by slowly treating Me_3CHMgI with about 0.5 the calcd. amt. of $BzMe$, boiling 2–3 hrs., distg. the Et_2O and heating the residue 4–6 hrs. on the H_2O bath (yield of crude product, b. 175–200°, 125 g. from 100 g. $BzMe$); 125 g. of this crude product heated to boiling in 50 cc. satd. HI and 30 g. red P, treated with I in small portions until fumes of HI began to escape through the reflux and boiled until the color of I disappeared gave trimethylethylbenzene, b. 186–8° (yield of crude product, h. 180–90°, 40 g.). The crude hydrocarbon (40 g.) in 24 cc. AcOH slowly dropped into 40 g. AcOH and 88 g. fuming HNO_3 at 35–40° and kept 3 hrs. at 50° yielded 30 g. *p*-trimethylethylnitrobenzene, b_D 152–4°, oxidized by $CrO_2 \cdot AcOH$ to *p*- $O_2NC_6H_4CO_2H$. *p*-trimethylethylaniline (16 g. from 23 g. of the NO_2 compd., 36 g. Sn and 75 cc. concd. HCl at about 100°), b_D 129–31°; 15 g. of this was diazotized, the gelatinous product added in small amts. to cold Na_2SO_4 , the reaction mixt. treated with Zn dust and AcOH to disappearance of the yellow color, the resulting sulfonate crystd. from H_2O (19 g. of plates, decomp. 190°) and converted by treating in a little boiling H_2O with HCl into the hydrochloride of A (needles perfectly stable when dry). The free A (9 g.), slightly yellow, h. 147–50°, soon solidifies completely, then m. about 60°, soon turns partly red in the air and liquefies in a few hrs.; in H decompn. occurs very slowly if at all. It forms hydrazones or osazones with sugars and reacts with HO acids such as *l*-erythronic, *d*-mannonic and *d*-gluconic, and forms a particularly stable and well crystd. compd., m. 160–3°, presumably the hydrazide, with *d*-galactonic lactone. The $Me_3CHCHMeC_6H_4NH_2$ (16.5 g.) with 17.5 g. *d*-hydroxymethylenecamphor gives 30.9 g. of a semicryst. product from dil. alc. (presumably a mixt. of equal parts of the isomers), with $[\alpha]$ 257.5° (0.6922 g. in 15.68 g. of 95% alc.), which when freed from the gummy material with ligroin and repeatedly crystd. from 95% alc., yields 8.2 g. of a compound, m. 168°, with $[\alpha]$ 300° (0.6540 g. in 20 cc. of 95% alc.); 40 g. of this, boiled 30 hrs. with 225 cc. of concd. HCl gives 8.6

g. B, b_{24} 139–40°, α 0.96° in a 1-dm. tube; *hydrochloride*, $[\alpha]_D^{20}$ 0.974 (1.0294 g. in 20 cc. H₂O containing 3 drops concd. HCl). The more sol. isomer of B could not be obtained cryst., the final *hydroxymethylenecamphor* product being a gum with $[\alpha]_D^{20}$ 229° (95% alc.), the *aniline* from which b_{24} 139–40° and showed α 0.76° in a 1-dm. tube; *hydrochloride*, $[\alpha]_D^{20}$ 0.565°.

CHAS. A. ROUILLER

Condensation of benzoyl chloride with ethylene in the presence of aluminium chloride. JAMES F. NORRIS AND HENRY B. COUCH. *J. Am. Chem. Soc.* **42**, 2329–32 (1920).—A comparison of the reactions of a number of compds. containing a Ph radical and analogous compds. containing a CH₂:CH radical having shown that the effect of these 2 groups was markedly similar, it appeared possible that a H atom in C₆H₄ and one in C₆H₅ might show similar reactivity, a possibility which was tested on C₆H₄ by a reaction which is possibly the most characteristic of aromatic hydrocarbons, *viz.*, condensation with acyl or alkyl halides in the presence of AlCl₃. As a matter of fact, when 25 g. BzCl, 24 g. AlCl₃, and 600 cc. CS₂ were boiled 2 hrs. and dry C₂H₄ was bubbled through the resulting suspension of crystals, a 25% yield of CH₂:CHBz was obtained. In view of these results and those of Krapivin (*C. A.* **5**, 1281), it seems highly probable that unsatd. compds. containing a double bond can replace aryl hydrocarbons in the Friedel-Crafts synthesis.

CHAS. A. ROUILLER

Reaction between phenylmagnesium bromide and the esters or anhydride of phthalic acid. LLOYD B. HOWELL. *J. Am. Chem. Soc.* **42**, 2333–7 (1920).—The differences in the results obtained by Guyot and Catel (*C. A.* **1**, 853) and by Shibata (*C. A.* **3**, 1172) on treatment of phthalic esters or the anhydride with Grignard reagents are due to the fact that they used different methods in isolating the products, the former crystg. from Et₂O or C₆H₆ the product prepd. from the di-Me ester and excess (over 3 mols.) of PhMgBr and obtaining triphenylhydroxy- α,α' -benzo- β,β' -dihydrofurfuran (A), m. 118°, while S., distg. *in vacuo* the product of the action of PhMgBr on the di-Et ester, obtained only the dehydration product (B) of A, m. 194°. H. has now found that when the di-Et ester is treated with 8 mols. PhMgBr and the viscous yellow oil obtained by decomp. the product with HCl, extg. with Et₂O and evapg. the Et₂O is allowed to stand several weeks, A gradually crysts. out, and on the other hand if the product from the di-Me ester and 5 mols. PhMgBr is distd. under 26 mm. B is obtained. Also, if A is slowly heated under 26 mm. up to 400°, it m. colorless 119°, begins to darken through yellow (160°), deep yellow (170°) and red-brown (200°) to dark amber above 350° (bath temp.); when the temp. of the vapor reaches 280°, moisture begins to condense in the cool parts of the app. and at 295° a pale greenish yellow fluorescent liquid begins to dist. over. If the heating is stopped at this point, the residue yields B on pressing upon clay and crystg. from PhMe.

CHAS. A. ROUILLER

Addition reactions of phosphorus halides. I. Mechanism of the reaction of the trichloride with benzaldehyde. JAMES B. CONANT AND ALEXANDER D. MACDONALD. *J. Am. Chem. Soc.* **42**, 2337–48 (1920); cf. *C. A.* **14**, 1827.—Just as the P in PCl₃ adds in the 1,4-positions in the conjugated systems of certain unsatd. ketones, becoming quinquevalent, so it adds in the 1,2-positions to simple aldehydes or ketones, the primary product being RCH(O.PCl₂)₂ (A). When, *e. g.*, BzH and PCl₃ are brought together

considerable heat is evolved and the addition reaction proceeds to a definite equil. With equimol. amts. of the 2 components only about 30% of A is formed and on treatment of the reaction mixt. with H₂O the A forms PhCH(OH)PO(OH)₂ (B) and the unchanged PCl₃ gives H₃PO₃ which can be detd. by cautiously oxidizing to H₃PO₄ and pptg. with magnesia mixt. Quant. expts. with varying amts. of the BzH and PCl₃ and also of an inert solvent (C₆H₆) showed that the primary addition reaction is reversible and can be represented by the equation $K = \frac{[BzH][PCl_3]}{V[BzH.PCl_3]}$, K being 6.9–8.3. PCl₃ reacts very slowly with Ac₂O below 50°. A rapidly, with formation

of AcCl and a mixt. of an acid chloride, $\text{PhCH}_2\text{O.POCl}$ (C), and an anhydride, $(\text{PhCH}_2\text{O.PO})_2\text{O}$ (D), so that when the original equil. mixt. is treated with Ac_2O the

equil. is disturbed and the reaction proceeds to completion; when a large excess of Ac_2O is used, most of the C is converted into D. The mixt. of C and D remains as a gum when the AcCl and excess of Ac_2O are evapd. off and the amt. of C can be detd. by treating with H_2O and titrating the HCl formed by the Volhard method. With 1 mol. each of BzH and PCl_5 the % of C in the resin was found to vary from 45 with 1 mol. Ac_2O to 6 with 2 mols. Ac_2O . That the resin is an intermediate product in the formation of B is shown by the fact that when boiled several hrs. with H_2O evapd. to dryness and treated in $\text{EtOH-Et}_2\text{O}$ with PhNH_2 it gives *aniline hydroxybenzylphosphonate*, $\text{PhCH}(\text{OH})\text{PO}(\text{OH})\text{ONH}_2\text{Ph}$, crystals from alc., m. $201-2^\circ$ (decompn.), which, treated with NaOH , distd. with steam, cooled, neutralized with AcOH , pptd. with $\text{Pb}(\text{OAc})_2$ and decompd. with H_2S , yields pure B, m. $170-2^\circ$. If the resin is dissolved in H_2O by gently warming and stirring, cooled and treated with the calcd. amt. of $\text{Ba}(\text{OH})_2$, it gives 65% of the *barium salt* (white powder only slightly sol. in H_2O and contaminated with $\text{Ba}_3(\text{PO}_4)_2$ of *anhydro- α -hydroxybenzylphosphonic acid* (E), $\text{PhCH}_2\text{O.POOH}$,

which could be obtained only in impure form (still containing about 10% Ba) by grinding the Ba salt with 6 N HCl , pouring off the liquid, washing with H_2O and drying *in vacuo* over H_2SO_4 ; it decolorizes KMnO_4 only $1/2$ as fast as B, forms no cryst. PhNH_2 salt and is converted into B by warming on the H_2O bath with concd. HCl until a clear soln. results; the hydrolysis into B can be followed by detg. the increase in acidity of the soln. of the Ba salt in H_2O on boiling. In H_2O hydrolysis is 36% complete in 3.5 hrs.; in 0.033 N HCl 49% in 3 hrs. and 67% in 16 hrs.; in N HCl 45% in 1 hr. and 66% in 8 hrs. It is suggested that this new class of acids with a $-\text{P.O}-$ ring be called "phos-tonic acids" from their obvious similarity to the lactonic acids. Hydroxyphosphonic acids can be most satisfactorily prepd., without using a large excess of either the aldehyde or PCl_5 , by cautiously mixing the aldehyde and a 10% excess of PCl_5 at $20-40^\circ$, letting stand 1-2 hrs., then adding 3-4 mols. glacial AcOH and keeping 1-2 hrs. longer at $30-35^\circ$, pouring into H_2O , evapg. to dryness and pptg. as the PhNH_2 salt from $\text{EtOH-Et}_2\text{O}$; yield, 72% in the case of B. E is first formed but slowly changes into the B; this change is accelerated by satn. with HCl .

CHAS. A. ROULLER

Pyrimidines. LXXXIX. Condensation of benzamidine with ethyl γ -diethoxy-acetoacetate. TREAT B. JOHNSON AND LOUIS A. MIKESKA. *J. Am. Chem. Soc.* 42, 2349-65 (1920); cf. *C. A.* 13, 2031.—If $(\text{EtO})_2\text{CHCOCH}_2\text{CO}_2\text{Et}$ (A) should react with amidines as it does with $\text{CS}(\text{NH}_2)_2$ (*C. A.* 9, 2655), it should give a new type of cyclic amine compds. which ought to have very promising pharmacological properties. In the present paper is reported the behavior of $\text{PhC}(\text{NH})\text{NH}_2$ (B) toward A. Three compds. are formed, depending on the conditions. When 10 g. of A and 7.15 g. B HCl , each in the calcd. amt. of 10% NaOH , are allowed to stand together at room temp., about 48 hrs., there seps. 3 g. γ -diethoxy- β -benzamidinocrotonylbenzamidine, $(\text{EtO})_2\text{CHC}(\text{NHCPH:NH})\text{:CHCONHCPH:NH}$ (C), prisms from alc., m. 106° , resolidifies and m. again 136° , insol. in alkalies, sol. in acids. The alk. filtrate from C, exactly neutralized with HCl , yields 2-phenyl-4-diethoxymethyl-6-oxy-pyrimidine (D), slender needles from 95% alc., m. 175° , converted by digestion with dil. HCl into 2-phenyl-6-oxy-4-aldehydopyrimidine (E), crystals from H_2O or alc., m. 205° , containing 1 H_2O which is so firmly held (it cannot be expelled at 135°) that it is assumed to be H_2O of constitution and the compd. is hence assigned the structure $\text{N:CPh.MH.CO.CH:CCH-}$

$(\text{OH})_2$. If, however, to 7.15 g. B HCl in 10% NaOH (1.83 g. NaOH) is added 1 mol.

A yellow oil begins to sep. at once and solidifies on stirring; after 48 hrs. it is crystd. from 95% alc., yielding γ -diethoxyacetylbenzamidine $(EtO)_2CHCOCH_2CONHCPh$: NH, extremely sol. in NaOH, m. 145°, rearranging at this temp. into D, m. 174°, also when allowed to stand 12 hrs. or heated a few min. with 10% NaOH. E in boiling H_2O with 1 mol. $NH_2OH \cdot HCl$ gives the *oxime*, crystals from alc., m. about 268° (decompn.), reduced by $SnCl_2$ in HCl to 2-phenyl-4-aminomethyl-6-oxypyrimidine hydrochloride, crystals from 95% alc., m. 263-5° (decompn.). CHAS. A. ROUILLER

Mercury derivatives of phthaleins. EDWIN C. WHITE. *J. Am. Chem. Soc.* 42, 2355-66(1920).—The results obtained in the lab. and the clinic in the treatment of genito-urinary infections with some of W.'s org. Hg compds. (*C. A.* 14, 3470) have been of sufficient value to warrant a description of their prepn. and properties. Of the compds. described in this paper only 1 is cryst. The others are amorphous and their chem. identification depends on a knowledge of the parent phthalein, detn. of the Hg content and, in some cases, of the 2nd group attached to the Hg atom. None show m. ps. In other words, the chem. data can establish only identity of compn. as between 2 substances of this class and not possible differences such as isomerism or variation in the state of mol. aggregation. Differences of this kind have a fundamental bearing on the biological behavior of dyes and the physico-chem. characteristics of a dye are almost as important as their purely chem. ones when it is to be used for pharmacological study or for clinical administration. In their work, W. and his collaborators have considered as different individuals any 2 substances which, though of the same compn., are not made by precisely the same procedure. The generic name "mercurochrome" has been assigned to all these Hg dyes. The position of the Hg has not been rigidly demonstrated but it is presumed to enter the phenolic residues in the *o*-positions to the HO or quinone O; 4, 2 or 0 atoms of Hg can be introduced according as there are 4, 2 or 0 free *o*-positions. The introduction of Hg has, apparently, qual. the same effect, but weaker, on the color as halogens. For the introduction of the Hg the dye may be used either in soln. as the free acid in alc. or as the salt in H_2O or in suspension in H_2O , and the Hg in insol. form as the oxide or in soln., preferably as the acetate. With the latter in alc. the product is an acetoxymercury deriv., $RHgOAc$, but in H_2O suspension there is partial hydrolysis and the product is a mixt. of acetoxy- and hydroxymercury compds. In the former class of compds. the AcO content was detd. by Brieger and Schuleman's method (*C. A.* 8, 1752). The Hg was detd. by treating the substance in hot H_2SO_4 with small amts. of $KMnO_4$, destroying the excess of $KMnO_4$ with $(CO_2H)_2$ and pptg. the Hg with H_2S . The following compounds were prepd. (the numbers in parentheses represent the calcd. and found % of Hg, resp.): $C_{20}H_{18}O_4HgOH$ (37.3, 35.3), from 3 g. phenolphthalein in 25 cc. *N* NaOH and 150 cc. H_2O boiled 3 hrs. with 3 g. yellow HgO , pptd. by CO_2 as a slightly purplish milky ppt., insol. in all the usual solvents except glacial AcOH, gives about the same color with alkalis as phenolphthalein. $C_{20}H_{18}O_5Na(HgOH)_2$ (A) (49.6, 49.9), from 200 cc. of 0.1 *N* NaOH satd. with about 3.5 g. fluorescein and boiled 4 hrs. with 5 g. HgO , with occasional additions of H_2O , forms an iridescent green scaly product when dried on the H_2O bath. $C_{20}H_{11}O_5HgCl$ (Cl, calcd. 6.26, found 6.28), from fluorescein with 1 mol. HgO and subsequent treatment with dil. HCl. $C_{19}H_{10}O_5SNa(HgOH)_3$ (58.7, 57.4), from 5 cc. of *N* NaOH dild. to about 150 cc. satd. with about 2 g. phenolsulfonephthalein and boiled 4 hrs. with 6 g. HgO , purple powder with bronze luster. $C_{20}H_{11}O_4(HgOAc)_3$ (55.0, 55.8), from 3 g. phenolphthalein in 50 cc. alc. and a filtered soln. of 25 g. $Hg(OAc)_2$ in 50 cc. H_2O and 50 cc. AcOH heated 3 hrs. on the H_2O bath and allowed to stand overnight, crystals losing 1 mol. AcOH (from 1 of the AcO and 1 of the HO groups) at 120°, sol. in NaOH with deep purple color, decolorized by CO_2 . $C_{20}H_9O_5(HgOH)_4$ (about 62.2%) and $C_{20}H_9O_5(HgOAc)_4$, from 3.3 g. fluorescein in 200 cc. of 0.1 *N* NaOH and 5 cc. AcOH boiled 10 hrs. with 25 g. $Hg(OAc)_2$ in 200 cc. H_2O and a little AcOH (yield, about 10 g.), sol. in

alkali with a color like that of A, giving with I the brilliant color of erythrosin. $C_{27}H_{18}O_4(HgOAc)_2$ (46.6, 47.4), from 3 g. *o*-cresolphthalein in 50 cc. alc. heated overnight on the H_2O bath with 25 g. $Hg(OAc)_2$ in 50 cc. H_2O and 50 cc. $AcOH$, sol. in alkalis with brilliant purple color. *Dibromohydroxymercurofluorescein* (*mercurochrome* 220), $C_{20}H_{12}O_6Br_2HgOH$ (28.4, 28.3), from 49 g. dibromofluorescein in 8 g. $NaOH$ and 50 cc. H_2O dild. to 200 cc., pptd. with 12.5 cc. $AcOH$, treated with a filtered soln. of 22.5 g. HgO in 25 cc. $AcOH$ and 50 cc. H_2O dild. to 100 cc., dild. to about 500 cc. and boiled until a filtered sample gave no test for Hg with $(NH_4)_2S$ (4.5-6 hrs.) (yield, almost quant.), is a red powder showing marked electrical properties when rubbed, sol. in 2 mols., $NaOH$ with deep-cherry-red color; the soln. is fairly stable but on long standing may deposit a small amt. of Hg or $HgOAc$; the free acid dissolves completely in $(NH_4)_2S$ and deposits HgS only slowly. Phenolsulfonephthalein (3.5 g.) in 200 cc. of 0.1 *N* $NaOH$ and 10 cc. glacial $AcOH$ boiled 3 hrs. with 15 g. $Hg(OAc)_2$ in 100 cc. H_2O gives a mixt. of $C_{19}H_{17}O_6S(HgOH)_4 + C_{19}H_{17}O_6S(HgOAc)_4$ (found Hg 62.1, $AcOH$ 6.2), dark brown powder slightly sol. in $MeOH$ with slight, in $AcOH$ with intense purple-red, in dil. $NaOH$ with bluish purple color.

CHAS. A. ROUILLER

Optical rotation, optical isomerism and the ring electron (ALLEN) 3. Disodium phosphate as a catalyst for the quantitative oxidation of glucose to carbon dioxide with hydrogen peroxide (WITZEMANN) 11A. Advances in the chemistry of terpenes and essential oils (RECLAIRE, ROCHUSSEN) 17. Normal vapor pressure (RECHENBERG) 2.

COHEN, JULIUS B.: *Theoretical Organic Chemistry*. New Ed. New York: The Macmillan Co. 604 pp. \$2.50.

COOK, E. P.: *Laboratory Experiments in Organic Chemistry, Designed Especially for Use with Stoddard's Introduction to Organic Chemistry*. 2nd Ed. Philadelphia: P. Blakiston's Son & Co. 70 pp. \$1. For review see *J. Frank. Inst.* 191, 140(1921).

Acetaldehyde. H. DREYFUS. U. S. 1,361,974, Dec. 14. In the manuf. of AcH by passing C_2H_2 into a soln. containing H_2SO_4 and Hg compds., an app. of $Fe-Si$ alloy is used which is both resistant to dil. boiling H_2SO_4 and to the action of Hg .

Acetaldehyde. M. SOLLER, J. L. R. HOTZ and BRITISH CELLULOSE & CHEMICAL MANUFACTURING Co. Brit. 151,086, June 14, 1919. The manuf. of AcH by passing C_2H_2 into H_2SO_4 solns. containing Hg compds. is carried out in app. whereof the parts exposed to acid and Hg are of Pb which has been coated electrolytically with PbO_2 . Details of the production of the coating are given. Cf. 105,064 (*C. A.* 11, 2138).

Aromatic amines. J. TCHERNIAC. Brit. 150,412, May 29, 1919. Aromatic nitro compds., particularly oxynitro compds., are reduced by treatment with HI in HCl soln. To reconvert the I produced into HI , such an agent as P may be added. The prepn. of *p*-aminophenol is described.

Camphor. J. H. BREGENT. Brit. 150,654, May 7, 1920. Camphor is recovered from the vapors which are produced in the manuf. of incandescent gas mantles, celluloid, photographic films, etc., by the use of phenol or its homologs, *e. g.*, crude cresol, as an absorbent. The camphor is liberated from the phenolic solvent by steam distn. or by pptn. with soda. Cf. 127,309 (*C. A.* 13, 2428), 128,640 (*C. A.* 13, 2983), and 131,938 (*C. A.* 14, 326).

Alkylene cyanohydrins. RÖHM & HAAS. Brit. 150,708, Sept. 1, 1920. Alkylene cyanohydrins are prepd. by treating alkylene halohydrins with aq. cyanide solns. while cooling to prevent decompn. of the product; in an example, ethylene cyanohydrin is prepd. from ethylene bromohydrin and aq. KCN .

Trichlorohydrin. H. F. SAUNDERS and L. T. SUTHERLAND. U. S. 1,362,355,

Dec. 14. Trichlorohydrin is prepd. by adding Cl to propylene chloride in the presence of a gaseous S compd. such as SO_2 , SO_2Cl_2 or a S chloride which acts as a catalyst, at about room temp. or higher. Sunlight or other actinic light preferably is employed to promote the reaction.

Urea. Soc. d'ÉTUDES CHIMIQUES POUR L'INDUSTRIE. Brit. 151,596, Sept. 2, 1920. Urea, or salts thereof, are obtained by gradually adding a powdered cyanamide, such as CaCN_2 , to an acid which is vigorously stirred throughout the addition; suitable acids are H_2SO_4 , H_3PO_4 or CO_2 .

Catalytic hydrogenation. DAYTON METAL PRODUCTS CO. Brit. 150,991, May 7, 1919. Relates to improvements in app. for the continuous catalytic hydrogenation of volatile substances at fairly high temps. and pressures. The improvements comprise: (1) a reaction chamber surrounded by a number of heating devices separately controlled; (2) arrangements for heat-insulating the whole or a part of the reaction chamber; (3) a return pipe for unconverted H, in conjunction with condensers for returning non-hydrogenated material to the reaction chamber and for the removal of the hydrogenated material, and (4) a removable cartridge for carrying the catalyst. The app. is stated to be specially applicable to the production of cyclohexane from benzene. The vapor of the material to be hydrogenated is mixed with H in a chamber and introduced through the pipe into the reaction chamber containing the catalyst. The products of the reaction escape by the pipe to a condenser, unconverted H being returned to the reaction chamber by a pipe. A suitable construction is specified. Cf. C. A. 14, 747.

Catalytic oxidation of anthracene. J. M. WEISS and C. R. DOWNS. U. S. 1,355,098, Oct. 5. A mixt. of anthracene vapor and air is passed through tubes heated to 300–500° and containing V oxide deposited on crushed pumice, to produce anthraquinone. O gas alone may be used instead of air or may be mixed with a proportion of N or other inert gas. Control of the reaction is effected by regulation of the temp., pressure, use of inert diluent gases and character of catalyst.

Nitration process. A. E. HOLLEY and O. E. MORR. Can. 206,337, Dec. 7, 1920. The material to be nitrated and the acid travel in opposite directions through a plant which comprizes continuously stirred regions each of which is in direct communication with an unstirred region.

II—BIOLOGICAL CHEMISTRY

HATTIE L. HEFT, EDGAR G. MILLER, JR. AND WILLIAM J. GIES

A—GENERAL

FRANK P. UNDERHILL.

Colloid-chemical bases of enzyme kinetics. A. FODOR. *Kolloid-Z.* 27, 242–9 (1920).—To visualize the intricacies of enzyme action, it should be treated rather "with mathematics" than "as mathematics." Expts. with a pure enzyme hydrosol prepd. from yeast ext. and capable of splitting polypeptides, indicate that the mols. of the substrate diffuse to the surface of the heterogeneous enzyme phase where it forms an evanescent compound; so that we must consider not only the "remaining quantity" of the substrate, but also its diffusion speed, which in turn depends on its adsorption balance. As Abderhalden and F. have shown (C. A. 13, 2883, 2884) the adsorption isotherm $C_1 = K.C_2^{1/n}$ is a special case of the more general formula $C_1 = K.C_2$. Where the exponent of C_2 is fractional, the change follows a logarithmic curve, thus simulating an autocatalytic process. For such systems the term *melakinetik systems* is proposed. In some cases the substrate may adsorb the enzyme, which has the effect of making $n > 1$ in the adsorption formula. The splitting

of a polypeptide by the negatively charged yeast enzyme is explained on the Ostwald theory of catalyzers. The following definition is suggested: Insofar as concerns physico-chem. relations, enzymes are products of colloidal nature, at present of plant or animal origin, whose activity is conditioned by their colloidal condition, *i. e.*, that of their sols. They form adsorption compds. with their substrates, losing thereby some of their surface energy, which brings about a hydrolytic splitting. The colloid-chem. factors must be considered as well as electrolytic phenomena. J. ALEXANDER

A great Belgian savant. C. JONAS. *J. Pharm. Belg.* 2, 941-4(1920).—A short biography of Jules Bordet, recently awarded the Nobel prize for his work in the medical sciences.

A. G. DuMEZ

Disodium phosphate as a catalyst for the quantitative oxidation of glucose to carbon dioxide with hydrogen peroxide. EDGAR J. WITZEMANN. *Rush Med. Coll., Chicago. J. Biol. Chem.* 45, 1-22(1920).—The work of Loeb and his coworkers (*C. A.* 5, 2110; 7, 492; 9, 1489) was repeated and confirmed. Further investigation proved definitely that the destruction of glucose is really an oxidation, proceeding practically completely to CO_2 and H_2O . The reaction is not a function of the H-ion concn. but is specific for Na_2HPO_4 . The mixt. is, apparently, not "poisoned" by the products of the reaction and the same Na_2HPO_4 may be used repeatedly. No evidence of the formation of a hexose-phosphate ester could be obtained. Since the rate of oxidation of glucose parallels that of the destruction of H_2O_2 in similar mixtures not containing glucose, it is probable that the reaction depends upon the formation of a highly reactive perphosphate. NaOH and Na_2CO_3 diminish the oxidation of glucose in Na_2HPO_4 - H_2O mixt., roughly in proportion to their action in decomp. H_2O_2 . Na_2HPO_4 does not catalyze the oxidation of glucose by air.

I. GREENWALD

The recovery of transmissivity in passive iron wires as a model of recovery processes in irritable living systems. II. RALPH S. LALLIE. *Clark Univ. J. Gen. Physiology* 3, 129-43(1920); cf. *C. A.* 14, 3682.—When passive iron wires are activated following long treatment with HNO_3 (d. 1.42), they revert spontaneously to the passive state after a temporary reaction which is transmitted rapidly over the whole length of the wire. The duration of this reaction at any region decreases with an increase in the concn. of acid above a critical concn. of 52-54% by vol.; in acid of 50% or lower, the reaction continues till all the metal is dissolved. Immediately after repassivation, the wire when again activated transmits but a short distance. It recovers this ability to transmit at first slowly, then more rapidly and eventually, after an interval which varies with the temp. and concn. of acid, the activation wave is transmitted an indefinite distance along the wire. This "complete recovery time" is nearly proportional to the excess of concn. of acid above 53-54%. The rate of recovery of transmissivity has a temp. coeff. similar to that of most chem. reactions and also to that of the rate of recovery of irritable living tissues after stimulation. In the process of recovery, two definite phases are distinguishable: (1) the redeposition of the continuous passivating surface layer of oxide or O compd.; (2) the progressive change of the newly passivated wire from the state of incomplete to that of complete transmissivity. The first phase is of brief duration, indicated by a sudden change of the elec. potential of the wire from that of active to that of passive iron. In the second and more prolonged period, the passivating film undergoes an alteration which consists in a progressive thinning till a minimal thickness of the passivating film (probably 1 molecule) is attained. Further thinning is prevented by local electrochem. oxidation. The phenomena of partial or limited transmission during the 2nd phase of the recovery process corresponds closely with the phenomena of conduction with decrement in tissues such as a nerve fiber. Other analogies with the behavior of irritable tissues as shown in threshold phenomena, distinction between "local" and "propagated" effects, summation and effects resembling electrotonus are described. C. H. RICHARDSON

A theory of injury and recovery. I. Experiments with pure salts. W. J. V. OSTERSHOOT. Harvard Univ. *J. Gen. Physiology* 3, 145-56(1920).—A continuation of studies on *Laminaria* (cf. *C. A.* 14, 3681). This paper is largely concerned with a mathematical treatment of exptl. data. Equations are developed which enable one to predict the elec. resistance of the tissues of *Laminaria* during exposure to NaCl or CaCl₂ and also the recovery curves after any length of exposure to solns. of these salts.

CHAS. H. RICHARDSON

The significance of the hydrogen-ion concentration for the digestion of proteins by pepsin. JOHN H. NORTHROP. Rockefeller Inst. *J. Gen. Physiology* 3, 211-27 (1920).—The rate of digestion of proteins by pepsin and the cond. of protein solns. are closely parallel. "If the isoelec. point of a protein is at a lower H-ion concn. than that of another, the cond. and also the rate of digestion of the first protein extends further to the alk. side." For solns. of ge'atin, the optimum H-ion concn. for the rate of digestion and degree of ionization is the same. If, to a protein soln. containing the optimum amt. of acid, a salt with the same anion as the acid is added, a depressing effect on the digestion of the protein by pepsin is observed; the same effect is obtained on the addition of an equiv. of acid. These facts support the hypothesis that the detg. factor in the digestion of proteins by pepsin is the amt. of ionized protein present in the soln. Such a hypothesis cannot be extended to all enzymes since in many cases the substrate does not exist in an ionized condition. The equil., however, might be between tautomeric forms of the substrate, only one of which is attacked by the enzyme. Cf. *C. A.* 13, 2041; 14, 2346, 2647.

CHAS. H. RICHARDSON

Ion series and the physical properties of proteins. II. JACQUES LOEB. Rockefeller Inst. *J. Gen. Physiology* 3, 247-69(1920); cf. *C. A.* 14, 3682.—The Hofmeister ion series does not give the correct expression of the relative effect of ions on the swelling of gelatin. Chlorides, bromides and nitrates do not have hydrating effects, nor do acetates, tartrates, citrates and phosphates have dehydrating effects on gelatin. At the same p_H , the effect on swelling is the same for the following gelatin salts: gelatin chloride, nitrate, trichloroacetate, tartrate, succinate, oxalate, citrate and phosphate, but is considerably less for the sulfate. This is the expectancy on the basis of the combining ratios of the acids with gelatin. Since with weak dibasic acids the anion in combination with gelatin is univalent while with H₂SO₄ it is bivalent, it is concluded that the valency and not the nature of the ion affects the degree of swelling. Expts. with alkalis supported this conclusion. Expts. on the relative soly. of different gelatin salts in alcohol-water solns. show the same influence of valency of the ion in combination with gelatin as observed in swelling. The drop in the curves for swelling, osmotic pressure or viscosity of gelatin which occurs at p_H 3.3 or slightly less is not due to a reduction in the concn. of ionized protein in the soln. The difference between the phys. properties of gelatin sulfate and gelatin chloride is not due to differences in the degree of ionization of the 2 salts.

CHAS. H. RICHARDSON

Spectral properties of tetanus toxin. FRED VLÉS. *Compt. rend.* 171, 524-6 (1920).—The method consisted in establishing by spectrophotometric measurement a curve of absorption with coeff. $K_1 = [\text{colog } I \div I_0] \div cl$ (in which I and I_0 are the intensities of the radiation, l the thickness of the absorbing layer and c its concn.) for the toxic complex (houillon-toxin) in the entire ultraviolet region. After some chem. or physical reaction has taken place in the medium a second absorption coeff. curve K_2 is detd., $K_1 - K_2 = f\lambda$, which is by difference the spectrum of the substance eliminated by the reaction, or that of a modified physical state. Observations were made upon the tetanus toxin, the same after prolonged heating at 65° and after the addition of the sp. antitoxin. Possibilities are suggested by the exptl. results but no conclusions given. Direct obtaining of the differential spectrum of toxin bouillons. *Ibid* 552-5(1920).—With a direct method the results obtained on heating the toxic

bouillon were the same as those given in the preceding paper. The modification of the spectrum of the toxic bouillon by the addition of antitoxin is not instantaneous and is a function of the time. It appears probable that there is a relation between the absorptive powers of toxic tetanus bouillons mixed with antitoxic serums, which varies with the antitoxic powers of the mixts.

L. W. RIGGS

The globulin of the cohune nut, *Attalea cohune*. CARL O. JOHNS AND C. E. F. GERSDORFF. U. S. Dept. Agr. *J. Biol. Chem.* 45, 57-67(1920).—The protein of the cohune nut consists almost entirely of a globulin, with only a trace of an albumin. The globulin resembles that of the coconut. Upon analysis by Van Slyke's method, it yielded the following figures: amide N 7.50%, humin N adsorbed by $\text{Ca}(\text{OH})_2$ 0.84%, humin N in $\text{C}_2\text{H}_5\text{OH}$ ext. 0.11, cystine N 0.53% arginine N 30.87%, histidine N 2.61%, lysine N 7.94%, $\text{NH}_2\text{-N}$ of filtrate 47.87%, non-amino N of filtrate 2.28%. The values for free $\text{NH}_2\text{-N}$ were 3.16 and 3.48, av. 3.32%, which is a little less than half the lysine N.

J. GREENWALD

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B—METHODS AND APPARATUS

STANLEY R. BENEDICT

Colorimetric analysis. XIII. The determination of mercury in urine. W. AUTENRIETH AND W. MONTIGNY. Univ. Freiburg i. Br. *Münch. med. Wochschr.* 67, 929-21(1920).—According to the expected amt. of mercury 500-1000 cc. urine are used. The urine is heated after addition of 20-25 cc. concd. HCl and 5-15 g. KClO_4 under a reflux condenser over the free flame about 2 hours, until the fluid is nearly colorless. To the hot filtrate sodium acetate is added for 20 cc. HCl , 35 g. of the cryst. salt and 0.02-0.03 ZnCl_2 . H_2S is passed through for one-half hour. After standing overnight the ppt. is filtered off on asbestos with suction and washed several times with H_2S water. The ppt. with the asbestos is transferred to a small porcelain dish and stirred with 5 cc. hot dil. HCl in which a few crystals of KClO_4 are dissolved.

Cl goes off, the rest is removed with a little alc., whereby the fluid must become colorless. The fluid is filtered into a small graduate, the asbestos is washed out repeatedly with small amts. of hot water. The total filtrate should not be more than 10-20 cc. After shaking, 8 cc. clear and colorless filtrate is measured into a 10-cc. graduate, 1 cc. of a clear 1% gelatin soln. is added and 1 cc. H₂S water. The mixt. is shaken. After 3-5 min. the color of the brownish soln. is compared in the Autenrieth-Königsberger colorimeter with a similarly prepd. standard soln. of mercury. Pb, Cu, Ag, Bi and Sn should not be present. As does not interfere. Evapn. is to be avoided in mercury detns. The results are good.

S. AMBERG

Cholesterolemia. I. A study of methods, with especial reference to colorimetric determinations. JOH. FEIGL, Hamburg. *Z. exp. Med.* 11, 178-238(1920).—The colorimetric method may be used for quant. detns. Technical and reactive difficulties are numerous and are discussed in detail.

E. B. FINK

Comparative studies on respiration. XIII. An apparatus for measuring the production of minute quantities of carbon dioxide by organisms. MARIAN IRWIN. Harvard Univ. *J. Gen. Physiology* 3, 203-6(1920); cf. *C. A.* 13, 240, 2695; 14, 1354, 2651, 3682.—A modification of the method of Osterhout (*C. A.* 13, 231). C. H. R.

Indole test on the spinal fluid for rapid diagnosis of influenzal meningitis. T. M. RIVERS. *J. Am. Med. Assoc.* 75, 1495-6(1920).—The test was made by stratifying an ether ext. of the spinal fluid with a mixt. of Ehrlich's reagent 4 parts, abs. alc. 380, concd. HCl 80. An old rose color at the junction of the liquids indicates indole. Eleven out of 12 meningitic strains of *B. influenzae* studied were indole formers. The test was negative when the fluids were first drawn, but if the fluids were incubated several hrs. a faintly positive test was obtained. The addition of one drop of sterile blood to 10 cc. of spinal fluid before incubation hastened and intensified the reaction so that the result was often strongly positive within 3 hrs. With few bacilli and no blood the test was weakly positive in 12 hrs.

L. W. RIGGS

Easy method for the distinction and estimation of lactose and glucose in urine. A. P. MATHEWS. *J. Am. Med. Assoc.* 75, 1658-9(1920).—The basis of this method is to ferment the urine with a large amt. of yeast when the glucose is fermented and its reducing power destroyed while the lactose remains unaffected. To 10 cc. of urine in a test-tube is added one-fourth of a cake of Fleischmann's yeast and shaken until there are no lumps. The uncorked tube is then placed in a slanting position in a beaker of water at 40-43°. Bubbles of air will escape from the urine and yeast for a minute and cease if there is no fermentation. If glucose is present bubbles of gas begin to rise along the slanting top of the tube within 2 min., the amt. of gas depending upon the amt. of glucose. With less than 1% of dextrose the fermentation is hardly visible, but with 3% the fermentation is stormy. A qual. result is certain within 10 min. Quant. results are obtained by allowing the fermentation to proceed for 50 min., filtering off the yeast and testing the reducing power of the filtrate. The difference between the reducing powers before and after fermentation indicates the amt. of dextrose present. Lactose may be identified by its osazone after the destruction of dextrose.

L. W. RIGGS

New method for detecting lactic acid in gastric juice or other organic fluids. EMILIO PITTARELLI. *Bull. acad. med.* 84, 132-5(1920).—The method, applicable to various organic fluids, is based upon the change of lactic acid to acetaldehyde by permanganate in a neutral medium. The only substances other than lactic acid likely to be encountered by the physician in lab. examn. which give the reaction, are ethyl alc. and mannitol. Lactic acid will be converted into acetaldehyde by permanganate only in neutral sol.; in acid or alk. soln. the reaction proceeds too far. Neutrality is provided by the addition of a satd. sol. of MgSO₄. After the mixture of the test fluid, MgSO₄, and permanganate has stood for 2-3 hrs. the material is filtered

and the filtrate tested for acetaldehyde by the addition of phenylhydrazine-HCl, diazosulfanilic acid, and KOH or NaOH. A red color indicates acetaldehyde, which can be detected when present in 1 part in 70,000.

G. H. SMITH

C—BACTERIOLOGY

A. K. BALLS

Variation in agglutinability of bacteria associated with variation of cultural characters. T. J. MACKIE. *Brit. J. Exptl. Path.* 1, 213-7(1920). LOUIS LEITER

Practical method for detecting in schizomycetes the ability to fix free nitrogen. Preliminary note. ADOLFO CAUDA. *Stas. sper. agrar. ital.* 53, 70-80(1920).—Upon agar containing no nitrogenous compds. algae do not turn green even under the action of light so that by making successive transplantations upon this medium a colorless variety of alga is secured. *Chlorella vulgaris*, *Protococcus* and *Chlorococcum* were most sensitive to variations of N and to the reaction of the medium. When this de-colored, whitish variety of alga is placed where conditions for nitrogenous nutrition are convenient they regain their green color easily, the chloroplasts returning to their original normal state. By placing upon the same medium devoid of N the whitish alga and the microorganism suspected of being a fixator of N the alga will turn green in case this power of N fixation exists. Numerous exptl. tests with *Azotobacter chlorococcum* gave positive results.

ALBERT R. MERZ

The influence of various chemical and physical agencies upon *Bacillus botulinus* and its spores. I. Resistance to salt. ZAE NORTHRUP WYANT AND RUTH NORMINGTON. *Mich. Agr. Coll. J. Bact.* 5, 553-7(1920).—A large number of strains of *B. botulinus* are not inhibited by percentages of salt ranging from 1 to 10 when growing in a medium with an alkalinity of -0.5 (Fuller's scale). This may not signify that *B. botulinus* will survive 10% or less salt in pickling as the additional factor of acidity enters here, and this anaerobe is said to be injured by even a slightly acid medium (0.5 to 0.8.) The results seem to indicate that the salt in pickling solns. is not the inhibiting factor.

JOHN T. MYERS

Milk powder agar for the determination of bacteria in milk. S. HENRY AYRES AND COURTLAND S. MUDGE. U. S. Dept. Agr. *J. Bact.* 5, 565-88(1920).—Formulas for three agar media which contain skim milk powder are given. In addition peptone and meat ext. or else yeast ext. are used. With these media it is possible to obtain not only a total count but a count of acid, alk., and peptonizing colonies.

JOHN T. MYERS

Bacterial inhibition. I. Germicidal action in milk. WILLIAM H. CHAMBERS. Univ. Ill. *J. Bact.* 5, 527-41(1920).

JOHN T. MYERS

Time-saving bacteriological apparatus. M. J. PRUCHA AND F. W. TANNER. Univ. Ill. *J. Bact.* 5, 559-63(1920).

JOHN T. MYERS

The use of washed agar in culture media. S. HENRY AYRES, COURTLAND S. MUDGE AND PHILLIP RUPP. U. S. Dept. Agr. *J. Bact.* 5, 589-96(1920).—Reduction of the content of Ca and Mg is a possible explanation of higher counts on milk obtained with washed than with unwashed agar.

JOHN T. MYERS

Investigations of the germicidal value of some of the chlorine disinfectants. F. W. TILLEY. Bur. Animal Ind., Wash., D. C. *J. Agr. Research* 20, 85-110(1920).—The members of the Cl group of disinfectants included in this investigation were: chloramine T, Dakin's soln. (NaOCl), eusol (HOCl) and Cl. Compared on a basis of wt. of chloramine T as against wt. of Cl as NaOCl or HOCl or as Cl in aqueous soln., chloramine T is less efficient than the others, but if the comparison is made on the basis of available Cl it may be much more or less efficient according to the organism treated. The expts. upon *B. tuberculosis* indicate that the Cl disinfectants are worth very little so far as this organism is concerned. A more or less "selective action"

was noted on the part of the different disinfectants. Thus, chloramine T was found to be very effective against *Staphylococcus aureus* but exhibited little antiseptic value in the case of *B. pyocyaneus*. The results of the expts. upon anthrax spores show that the germicidal action of Cl compds. is not always so speedy as is commonly supposed but may extend over several days. The addition of NH_3 to solns. of Cl or hypochlorites very greatly increases germicidal activity and tends to prevent depreciation in value on the addition of org. matter. Owing to the great diminution in germicidal value on addition of org. matter as well as of their injurious effects on metals and fabrics, the Cl disinfectants, apart from their use in the treatment of infected wounds and the disinfection of drinking water, do not seem to be suited as a class for use under the usual conditions and by the usual methods of general disinfection. W. H. ROSS

Studies of the enzymes of pneumococcus. I. Proteolytic enzymes. O. T. AVARY AND GLENN E. CULLEN. Rockefeller Inst. *J. Exp. Med.* 32, 547-69 (1920).—Pneumococci contain an intracellular enzyme or enzymes which hydrolyze to some extent intact protein and hydrolyze with striking avidity peptones. The optimum reaction for hydrolysis is pH 7 to 7.8, which also represents the optimum for the growth of pneumococcus. Solns. of intracellular substance of comparable enzymic activity can be prepd. by dissolving the bacteria in bile, in Na choleate or by mechanical and autolytic disintegration of the cell. The rapidity with which the peptone is hydrolyzed is proportional to the concn. of the enzyme. The activity is destroyed by heating at 100° for 10 min. Increasing the pH to 5, the acid death point of the pneumococcus, suspends the activity but does not destroy the enzyme, for activity is restored by readjustment to pH 7.8. Attenuation of virulence to 0.000001 of the original virulence had no measurable quantitative effect on the enzyme activity. **II. Lipolytic enzymes: esterase.** *Ibid* 571-82.—Pneumococci contain an intracellular enzyme of marked lipolytic activity as measured by the acid liberated by its action on tributyrin. Solns. may be prepd. by dissolving pneumococci in bile. The optimum reaction for max. activity is pH 7.8. Heating at 70° for 10 min. destroys its activity. Attenuation of virulence of pneumococcus had no measurable effect on the enzymic activity. **III. Carbohydrate-splitting enzymes: invertase, amylase and inulase.** *Ibid* 583-93.—A method is described for the prepn. of an active enzyme-containing soln. of pneumococci, in which no living cells are present. These enzymes are capable of hydrolyzing sucrose, starch and inulin. The invertase and amylase are active within the limits of pH 5 to 8, with an optimum of about pH 7. The studies indicate that the enzymes described are not true secretory products of the living cell, but are of the nature of endoenzymes, since their activity can be demonstrated only when cell disintegration has occurred. C. J. WEST

D—BOTANY

CARL L. ALSBERG

Oligodynamic action of a metal in nature. RICHARD EMSLANDER. *Kolloid-Z.* 27, 254-5 (1920).—In Eichstätt, Bavaria, local limestone deposits furnish slate-like splittings used for roofing. On such roofs lichens furnish a characteristic patina, but bare stripes showing the white of the underlying limestone appear beneath bare copper wire, because copper in some form (probably colloidal) is washed down by the rain and is adsorbed by the lichen in sufficient quantity to inhibit its growth.

JEROME ALEXANDER

Respiration of cereal plants and grains. II. Respiration of sprouted wheat. C. H. BAILEY AND A. M. GURJAR. Minn. Agr. Expt. Sta. *J. Biol. Chem.* 44, 5-7 (1920); cf. C. A. 13, 2092.—“Respiration, and consequently the quantity of heat released per unit of time and material, proceeds at a higher rate in sprouted grain than in normal wheat.” **III. Respiration of rice paddy and milled rice.** *Ibid* 9-12.—With rice, as with wheat, the germ, or embryo, is the seat of the most active respiration.

The acceleration of respiration with increasing moisture is greater with rice than with wheat. IV. The respiration of frosted wheat plants. *Ibid* 13-15.—Plants that have been frozen produce more CO_2 than do others cut at the same time but not frozen. The difference grows less with increasing maturity, probably being due to the increasing desiccation. V. Note on the respiration of wheat plants infected with stem rust. *Ibid* 17-18.—"The rate of respiration of the infected plants was materially lower than the sound plants."

I. GREENWALD

Occurrence of diastase in the sweet potato in relation to the preparation of sweet potato sirup. H. C. GORE. U. S. Dept. Agr. *J. Biol. Chem.* 44, 19-20(1920).—"Sweet potatoes are high in diastatic power and it is possible to convert nearly all their starch into so' carbohydrates by slowly cooking the potatoes in H_2O (60-80° for 1 hr., then raising to 100° in 0.5 hr.). The pulp formed by mashing the cooked potatoes with hot H_2O drains readily, permitting the easy recovery of the sweet juice."

I. GREENWALD

Experimental studies on mitochondria in plant cells. N. H. COWORY. *Biol. Bull.* 39, 188-98(1920).—This study was made on the cells of the elongating part of the root cap of sprouting peas. After sprouting the plantlets were treated by the Regaud formalin-dichromate method, and sections (cut 4μ) were stained with Fe hematoxylin. It is necessary to use fresh formalin, any HCOOH present being neutralized by MgCO_3 . Plantlets were subjected to the actions of centrifuging, plasmolyzing agents such as cane sugar, deprivation of water 36 hrs., variations of light, variations of temp. from that of ice and salt to 73°, submergence in tap water, restriction of air space, CHCl_3 vapor, H_2O vapor, $\text{C}_2\text{H}_5(\text{OH})_2$, and lecithin. The results are shown in 18 figures. "Mitochondria are changed to an abnormal degree only under severe conditions which either kill the cell or render its recovery very improbable. Even when the cell has been killed its general appearance varies in almost every case, the mitochondria however are very much reduced in number or disappear entirely by apparent soln., or in the case of plasmolysis through the prior appearance of vesicles. In the expts. with submergence and restricted air space, when sufficient O for respiration has not been supplied, the mitochondria are changed in a somewhat similar manner but the location of the changes is reversed." Plantlets placed so that their radicles were in intimate contact for 24 hrs. with sphagnum moss soaked with a 1% soln. of lecithin, showed many remarkable changes. These cells were loaded with granules and filaments suggesting imbibition of the lecithin from the surrounding fluid and subsequent incorporation in the mitochondrial substance. The author hopes to make an intensive study of the influence of lecithin upon the mitochondria in plant cells.

L. W. RIGGS

Toxicity of iron and the antitoxic properties of copper to the ferrous salts. L. MAQUENNE and E. DEMOUSSY. *Compt. rend.* 171, 218-22(1920).—Ferrous compds. are more toxic to plants than ferric, hut owing to the oxidation which ferrous compds. readily undergo and the passage of ferric compds. into insol. basic substances with the liberation of acids, it is impossible to state the exact condition of the Fe in a culture liquid at any instant. Expts. were made with peas grown in sand in saucers with 10 cc. of culture liquid containing FeSO_4 or $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2$ for the ferrous, and $(\text{NH}_4)_2\text{Fe}_2(\text{SO}_4)_4$ for the ferric compd. The action of Fe was tested in the absence and in the presence of KH_2PO_4 , CaSO_4 and CuSO_4 , the expts. being arranged in series with suitable controls so as to test the action of each constituent added. Effects were estd. by measuring the length of root formed. In the absence of phosphate the presence of 0.5 mg. CaSO_4 greatly increased the growth until the Fe reached a concn. of 2 mg. per. 10 cc. when the action of CaSO_4 was slight. The presence of 50 mg. KH_2PO_4 and 8 mg. CaSO_4 greatly decreased the toxic action of the Fe. The addition of ferric alum to a culture soln. containing ferrous alum reduced the toxicity of the latter. Colloidal

Fe_2O_3 had very slight toxic action. Cultures in quartz tubes gave results similar to those made in saucers. CuSO_4 in the culture liquid reduced the toxicity of ferrous compds. 50% while it had practically no effect on ferric compds. These results suggest a new use for CuSO_4 in agr. especially on lands underlain with pyrites. L. W. RIGGS

Are vitamins necessary to the development of plants? AUGUSTE LUMIÈRE. *Compt. rend.* 171, 271-3(1920); cf. Agulhon and Legroux C. A. 13, 855; Mazé, C. A. 13, 3212.—Fresh beer yeast, very rich in vitamins and capable of rapidly curing polyneuritis in pigeons, when heated at 135° for 1 hr. loses all of its antiscorbutic properties for pigeons but retains properties which favor the development of fungi grown on a weak soln. of $(\text{NH}_4)_2\text{C}_4\text{H}_4\text{O}_6$ and $\text{C}_4\text{H}_8(\text{OH})_2$. It is concluded that vitamins are indispensable to life, and cannot be replaced by definite chem. compds. which are pptd. by phosphotungstic acid and the alkaloidal reagents. They are retained by filtration through fuller's earth and are destroyed by heat, when none of the properties capable of improving culture media can be recovered. L. W. RIGGS

Crystallizable sugar and free acids in vegetables. H. COLIN. *Compt. rend.* 171, 316-8(1920).—"All juices of plants have a hydrolyzing power inferior to that supposed by their concn. of free acid." If to a soln. of sucrose orange juice is added to make the acidity $N/120$ and the sucrose 5%, after 10 hrs. at 80° the rotation is changed from $6^\circ 45'$ to $2^\circ 45'$. A second soln. of sucrose with citric acid added to make the acidity $N/120$ and sugar 5%, after 10 hrs. at 80° shows a change in rotation from $6^\circ 44'$ to $-1^\circ 48'$. A third sample containing sucrose 5%, citric acid to $N/120$ and K citrate to $N/500$, after 10 hrs. at 80° shows a change in rotation from $6^\circ 40'$ to $2^\circ 12'$. Thus citric acid has very slight hydrolyzing power in the presence of citrates. This accounts for the action of orange juice which contains both citric acid and citrates. Hydrolyzing power of a plant juice depends upon its concn. in H ions. L. W. RIGGS

Utilizable experiments in vegetable physiology on osmosis and aspiration due to evaporation. PIERRE LESAGE. *Compt. rend.* 171, 358-60(1920).—Expts. of Dutrochet and of Askenasy upon osmosis were repeated by L. with variations in the membrane used, in the osmotic liquid and in the position of the membrane with reference to the liquid. Membranes of acetocellulose gave results comparable to those with bladder material when used upon solns. of sugar. With NaCl solns. in alc. the alc. appeared to modify the acetocellulose membrane making it less permeable to NaCl. The expts. are described with much detail, and possible applications to questions of animal and vegetable physiology are suggested. L. W. RIGGS

Appearance of alcoholic yeast in vineyards. F. GRENET. *Compt. rend.* 171, 411-2(1920).—"The alc. yeast appears in the vineyard only at the time of the ripening of the grape. Its distribution is attributed to a dipterous insect, *Drosophila melanogaster*. L. W. RIGGS

Remarks on the biochemical method of studying glucosides apropos of the note of P. Delauney. EM. BOURQUELOT. *Compt. rend.* 171, 423-5(1920); cf. following abstract. —B. gives a review with dates of the sepn. of glucosides hydrolyzable by emulsin beginning with salicin sepd. by Leroux in 1829 to scabiosin in 1920, 56 in all. This method has been applied to 281 species of phanerogamous plants in B.'s lab. Glucosides existed in 205 of these species. Deducting the 56 above, 149 of these glucosides remain to be isolated. Probably a similar proportion of plants containing glucosides will be found among other species of plants. L. W. RIGGS

Extraction of the glucosides of two indigenous orchids; identification of these glucosides with loroglossin. P. DELAUNEY. *Compt. rend.* 171, 435-7 (1920).—A glucoside was sepd. from *Orchis simia* Lam. and from *Ophrys aramifera* Huds. which gave the physical constants of loroglossin. Details for extra. purification and tests are given at length. L. W. RIGGS

Action of radiations of different wave lengths upon chlorophyll assimilation. RENÉ WURMSER. *Compt. rend.* 171, 820-2(1920); cf. *C. A.* 14, 2653.—It is generally admitted that the max. assimilation of chlorophyll in a green plant exposed to solar rays occurs in the vicinity of $\lambda = 680m\mu$, but the existence of a second max. at about $480m\mu$ is disputed. Seven concordant tests upon the green alga *Ulva lactuca* showed that if the velocity of assimilation, incident energy and absorbed energy under the condition of $\lambda > 580m\mu$ were severally represented by 100 the corresponding figures for $\lambda = 680-460m\mu$ were 24, 50 and 7, while for $\lambda / 460m\mu$ the figures were 80, 80 and 48. With the red alga *Rhodomenia palmata*, the velocity figures for the 3 different groups of wave lengths were 100, 50 and 18.

L. W. RIGGS

Biologic significance of alkaloids in plants. G. CIAMICIAN AND C. RAVENNA. *Compt. rend.* 171, 836-9(1920); cf. *C. A.* 14, 1133, 2810.—Expts. with young bean plants proved that NH_4 salts had no special toxic action but the 3 methylamines and tetramethylammonium salts were poisonous, the toxicity increasing with the number of CH_3 groups. Xanthine was non-poisonous, dimethylxanthine was toxic and trimethylxanthine still more toxic. This relation of alkyl groups to toxicity was studied with uric acid, pyridine, piperidine, coniine, nicotine, morphine, quinine, cocaine and many other closed-chain compds. It was found to be generally true that the introduction of alkyls or acyls increased the toxicity of the fundamental substance. Eight samples of a paste of spinach in the presence of O were severally treated with 2 g. of various alkaloids, pyrocatechol and guaiacol to test the action of enzymes upon the added substance. The amts. recovered ranged from 0 in case of morphine to 1.92 g. in the case of caffeine. The results showed that the compds. most toxic to bean plants are also those most resistant to the action of oxidizing enzymes. These results are discussed at length.

L. W. RIGGS

New color reactions for distinguishing mycologic species. J. BARLOT. *Compt. rend.* 171, 1014-16(1920).—Various fungi, particularly species of *Amanita*, gave colors with 20-40% soln. of KOH or NaOH (cf. *Compt. rend.* 170, 679), which were in some cases characteristic of the species. This paper records the results of study of the action of KOH upon *Mycena pura* (poisonous) and *Laccaria laccata* (edible), which are readily distinguished by the greenish yellow and dark brown color reactions. *Gomphidius viscidus* gives an intense violet-brown, but *G. glutinosus* a weak yellowish brown. Many other reactions are given. The active coloring principle of *Lactarius turpis* may be obtained as follows: macerate the fungus in dil. NH_4OH , filter, evap. filtrate; a brown amorphous residue remains, sol. in warm water from which the coloring matter is pptd. by $Pb(AcO)_2$ without combining with the Pb. The ppt. is collected, washed and dissolved in KOH soln. with violet or violet-red color. The coloring principle is sparingly sol. in acids giving a yellowish soln. with AcOH, carmine red with HNO_3 and violet-red with H_2SO_4 .

L. W. RIGGS

Metachromatin and tannin compounds of the vacuoles. PIERRE DANGEARD. *Compt. rend.* 171, 1016-9(1920).—Metachromatin is a substance which has been observed in the vacuoles of mushrooms, where it gives rise to the phenomena of vital staining and under certain conditions forms metachromatic corpuscles. Since this substance is found in algae and many phanerogams it is general in character. Histologic study shows that the young forms of vacuome contain metachromatin to which their vital staining is due. They have the appearance of a chondriome, but are distinguished from the latter by their evolution. Throughout their transformations they belong to the same formation as the vacuolar system. The impregnation by tannin is only an important stage in these modifications.

L. W. RIGGS

Variations in the composition of wheat. LINDET. *Compt. rend. agr. France* 6, 753-4(1920).—At the end of June 49% of the total N was sol., while by the end of July it fell to 10-14% and on germinating it rose to 36%. When the grain is forming

76% of the total phosphates are sol. and later only 35, then 42% are sol. The acidity is first 0.3 then 0.016 and finally 0.048. The sol. N content is of value to the baker.

F. M. SCHERTZ

The influence of the "thirst" period upon the growth of plants. TH. PFEIFFER, A. RIFFEL AND CHARLOTTE PFOTENHAUER. *Landw. Versuchsst.* 96, 353-63 (1920).—Plants which had a const. water content of the soil produced higher or at least no smaller yields than those with alternating thirst periods. Drying intervals cause injury which depends upon the species of plant and its habitat. The water requirement per g. of dry substance is always less when thirst periods are introduced and plants which inhabit dry climates show a greater gain in dry substances over those which require more moist conditions.

F. M. SCHERTZ

The formation of starch in the green plant. C. RAVENNA. *Gazz. chim. ital.* 50, I, 359-61 (1920); cf. C. A. 14, 2811.

E. J. WITZEMANN

The behavior of some organic substances in plants. XII. G. CIAMICIAN AND C. RAVENNA. *Gazz. chim. ital.* 50, II, 13-46 (1920); cf. C. A. 14, 2810.—The paper is composed of two parts of which the 1st deals with tests on the action of organic compds. of plants and the second with the autooxidations produced by plant enzymes (cf. C. A. 14, 1133). Part I. The details of the expts. are given and summarized in 4 tables and permit of extending the previous conclusions. That the introduction of alc. and acid radicals exalts the toxic action of the org. substances was confirmed. Isoamylamine (A) has a highly toxic action on kidney beans. Its action has now been compared with normal amines in order to observe the influence of the length of the chain. The toxicity is less in amylamine (B) than with EtNH_2 , in fact the former is really little toxic. But MeNH_2 is also less toxic than EtNH_2 . Moreover A is not only more toxic than B but gives rise to an albinism similar to that produced by nicotine. The greater toxicity of A is no doubt due to the 2nd Me group since K isobutyrate is also more toxic than K butyrate. Expts. with formamide and acetamide showed appreciable effect only with the former owing perhaps to the aldehyde group. Oxalic acid is a little more toxic than succinic acid, which is in harmony with action of the amines. In extending the observations with Me and K salicylates Me and Et tartrates were compared with K tartrate, which is harmless. Both were toxic: the Et more than the Me ester. Expts. with indole and methylindole were not conclusive. Results showing the slight toxicity of methylpyridine or picoline in comparison with the nontoxicity of pyridine confirm the influence of Me again. Similarly piperidine is less toxic than N-methylpyridine and coniine. Likewise α -methylquinoline is more toxic than quinoline or isoquinoline, which have about the same effect. Cocaine is strongly toxic; the toxicity of ecgonine is less and about the same as that of the Me ester of norecgonine; norecgonine however does not disturb the growth of the kidney bean plant. These results show that the influence of various radicals and side chains is indisputable, although the chem. mechanism by which they exercise their action in the organism is completely unknown. Unlike the salts of Me_2NOH those of betaine were but slightly toxic, which was unexpected. The expts. on the influence of the toxic substances on the formation of starch and its disappearance during the night were continued especially with theobromine and caffeine. With the latter substance the leaves appear pale, variegated with yellow, larger in size and give up to a certain point a more intense reaction with I. Some of the substances as was noted previously seem to cause darker color in the leaves that was thought not to be due to chlorophyll but which spectrographic examn. showed to be due to chlorophyll. The more intense I reaction was found to be due to a greater concn. of starch. Part II. In the preceding paper the transformations that some organic substances undergo in contact with spinach hash were described. The behavior of asparagine, lactic acid, salicylic acid, mandelic acid and menthone is now described. No essentially new facts were ascer-

tained but some facts were better established and some suppositions formerly advanced were excluded. The transformations that asparagine undergoes through the action of plant enzymes in the presence of O are due in part to oxidations by which AcH , AcOH , HCO_2H as well as propionic and succinic acids are formed. The formation of the latter is due to a fermentative process. Menthone by autooxidation in the presence of vegetable ferments produces besides the lower fatty acids small amts. of succinic acid, which shows that a more profound oxidation takes place than that produced by light. The expts. with lactic, salicylic and mandelic acids were done in order to see if the 1st 2 compds. give compds. saponif. by emulsin and dil. acids as was observed with mandelic acid. It was found that these 2 acids besides undergoing partial oxidation combine in some way so that Et_2O does not ext. them. This is also true of tartaric and succinic acids. The nature of these compds. could not be detd. It was supposed that they were of a glucoside nature since inoculation of maize with saligenin gives salicin, but this could not even be established again this year for mandelic acid. The negative results in attempts to ext. glucosides with AcOEt rendered the existence of such compds. improbable. Moreover the fact that treatment with acid gives nearly the whole of the mandelic acid used back again but no reducing substance (sugar) is further proof of the absence of glucosides. The compd. is probably a very labile one.

E. J. WITZMANN

Lignin and its reactions (KLASON) 10. The form of acidity of soils and its meaning in plant physiology (KAPPEN) 15.

CZAPEK, FR.: *Biochemie der Pflanzen*. II Vol. 2nd Ed. revized. Jena: G. Fischer. M. 66, bound M. 77.

E—NUTRITION

PHILIP B. HAWK

NORMAL

Chemistry of vitamins. ATHERTON SEIDELL. *J. Ind. Eng. Chem.* 13, 72-5 (1921).—A historical and descriptive review of the subject, with especial reference to the H_2O -sol. antineuritic vitamin. The exptl. procedures in vitamin work are briefly described. S. inclines to the view of Walsche (*C. A.* 13, 1069) that vitamins play the part of enzymes. He compares them as regards occurrence, stability towards heat and alkalis, soly. relations, dialyzability, loss of activity in fractionation procedures, and amts. necessary for causing transformations; and concludes that there is no particular reason why vitamins should not be classed for the present with enzymes.

A. L. BARKER

Relation of fodder to the antiscorbutic potency and salt content of milk. ALFRED F. HESS, L. J. UNGER AND G. C. SUPPLEE. New York City Dept. of Health. *J. Biol. Chem.* 45, 229-35 (1920).—Five Holstein cows that had been stall-fed throughout the winter on a ration of ensilage, hay and concentrates were fed, for 3 weeks, a daily ration of 25 lbs. of a mixt. of 1 part bean meal, 2 parts (flaxseed) oil meal, 2 parts hominy, 2 parts gluten meal and two parts bran, and 8 lbs. of kiln-dried beet pulp, 4 qts. molasses and 12 lbs. straw. After the first few days the cows ate practically all of this. The milk production fell from an av. of 40 lbs. to about 30 but then increased until at end of period it was 35 lbs. At that time a mixed sample was collected and dried "by means of the Just roller process, by which it is subjected to about 230° F. for a few seconds." The cows were then placed on pasture, a small amt. of concentrate being given for the first few days. The production of milk did not increase. At the end of 3 weeks, another sample of milk was collected and dried. For use in the feeding expts., the dry milks were then brought to the original vol. Guinea pigs receiving 80 cc. dry fodder milk developed scurvy in 21 days and died within 56 days. Those receiving pasture milk did not develop scurvy and were alive at end of 120 days. Since the guinea pigs receiving the dry fodder milk developed scurvy as rapidly as do those

receiving milk heated to 110° for 1 hr., it seems that the dry fodder milk was practically devoid of antiscorbutic vitamine. The prompt appearance of this substance in the milk when the cows were pastured also indicates that very little, if any, of this vitamine is stored in the cow's body. Cf. following abstr. Analyses of the milks showed no apparently important difference other than increase in citric acid of from 0.008% in dry fodder milk to 0.13% in pasture milk, in CaO from 0.138 to 0.165%, in P₂O₅ from 0.158 to 0.190%, in Cl from 0.054 to 0.097% and a decrease of SO₄ of from 0.023 to 0.014%.

I. GREENWALD

Vitamine studies. VI. The influence of diet of the cow upon the nutritive and antiscorbutic properties of cow's milk. R. ADAMS DUTCHER, C. H. ECKLES, C. D. DAHLE, S. W. MEAD AND O. G. SCHAEFER. Univ. Minn. *J. Biol. Chem.* 45, 119-32 (1920); cf. C. A. 14, 2505 and preceding abstract.—"Two cows, a Jersey and a Holstein, were placed upon a vitamine-poor ration (equal parts wheat middlings, gluten feed, ground oats and ground barley with roughage of equal parts chopped timothy hay and oat straw) in the middle of January. Mixed milk was fed in varying quantities to several groups of guinea pigs, new groups of guinea pigs being added from time to time as the expt. progressed. Beginning June 1, the cows were given a vitamine-rich ration (grain as before but turned out to pasture daily) and the former expt. was repeated. From the data obtained it was concluded that the vitamine content of cow's milk is dependent upon the vitamine content of the ration ingested by the cow. Twenty cc. of summer milk were superior in nutritive value and in antiscorbutic potency to 60 cc. of winter milk. It was found that there is a tendency for the milk to become poor slowly, when the diet of the cow is low in vitamins, while the milk becomes of higher nutritive value almost immediately upon the ingestion of a vitamine-rich ration."

I. GREENWALD

Growth on diets poor in true fats. THOMAS B. OSBORNE AND LAFAYETTE B. MENDEL. Yale Univ. and Conn. Agric. Expt. Sta. *J. Biol. Chem.* 45, 145-52 (1920).—Three rats were fed a dried mixt. of starch paste (equiv. to 72 parts starch), 23 parts meat residue (C. A. 12, 284) and 5 parts salt mixt., *ad lib.* They also received, daily, a mixt. of 0.4 g. brewery yeast, 0.2 g. alfalfa and 0.2 g. cane sugar. Three other rats received a similar diet except that the meat residue was first extd. 5 times with Et₂O containing a little EtOH. The rats grew as rapidly as on the normal diet. "We cannot avoid the conclusion that if true fats are essential for nutrition during growth the minimum necessary must be exceedingly small."

I. GREENWALD

Vitamine requirements of the rat on diets rich in protein, carbohydrates and fat respectively. CASIMIR FUNK AND HARRY E. DUBIN. *Science* 52, 447-8 (1920).—In a feeding test with pigeons the diets were made up of starch, sugar, casein and fat for the first test in the resp. proportions of 60, 12, 12 and 12; for the second test 12, 60, 12, 12; for the third 12, 12, 60, 12; and for the fourth 12, 12, 12, 60. Four parts of salts were added to each diet. The onset of beriberi occurred in 24, 28, 30 and 40 days resp. This expt. was checked by feeding rats 80 days with meat, sugar, starch and lard, the diet in the first expt. being 49, 12, 12, 12 resp., and alternated in the subsequent tests as with the pigeons. Three parts of salts were added to each diet. The meat, sugar, starch and lard were tested and found to be free from "vitamine B." Rats were fed on these diets for 25 days when it was found that those on a meat diet did not require the addition of extra vitamine (autolyzed yeast) while those fed the other rations made such a small gain, or even loss in case of the lard diet, that yeast ext., orange juice, cod-liver oil and agar were fed to a part of each group of rats. The gains in wt. in per cents for the 25 and 55-day periods, resp., were for meat diet 43 and 86, sugar 15 and 55, sugar with vitamine 15 and 145, starch 6 and 62, starch with vitamine 6 and 165, lard 9 loss and 8 loss, lard with vitamine 9 loss and 34 gain. In general rats on a protein diet and those on other diets but receiving extra vitamine looked healthy.

but the others did not, the evidences of improper nutrition were plainly shown. "The findings reported here show conclusively that although the qual. food requirements of a well balanced diet have been pretty well established, this cannot be said of the quant. relationship between the dietary constituents necessary for proper growth." The question of pellagra and war edema being avitaminoses is left open. L. W. RIGGS

Influence of dry versus fresh green plant tissue on calcium metabolism. E. F. ROBB. Univ. Minn. *Science* 52, 510(1920).—R. quotes McIlanby to the effect that the antirachitic vitamine, which is the same as fat-sol. A, is not destroyed by drying, while the antiscorbutic vitamine is greatly reduced except in acid foods. This is contrary to the conclusions of Hart, *et al.* (cf. C. A. 14, 3707). In R.'s expts, guinea pigs that had been on a diet of dried plants 14 or 21 days before the exptl. period, eliminated twice as much Ca as those that had been on a green diet. Scurvy appeared in all animals on the dry diet. R. suggests that the loss of Ca in the expts. of Hart *et al.*, may have been due to scurvy. L. W. RIGGS

ABNORMAL.

The use of a high-fat diet in the treatment of diabetes mellitus. L. H. NEWBURGH AND PHIL L. MARSH. Univ. Mich. *Arch. Intern. Med.* 26, 647-62(1920).—The use of diets containing about 9% of the calories as protein, about 7% as carbohydrate and the remainder as fat, is recommended. I. GREENWALD

Fat metabolism in diabetes mellitus. NATHAN F. BLAU AND SAMUEL T. NICHOLSON, JR. Clifton Springs Sanitarium, N. Y. *Arch. Intern. Med.* 26, 738-50(1920).—The changes in the blood of 26 patients undergoing the Allen-Joslin treatment were studied. Of these 11 showed an increase in the blood lipoids with decreased sugar. Clinically, these were all severe cases of diabetes. Blood lipoids rose and fell with blood sugar in 7 cases, the change in the former being slower than that in the latter. All but 2 of these were in a milder stage of the disease. In 8 cases there was an almost const. value for the blood lipoids, at high level (total fats about 1%; cholesterol, whole blood about 0.25%, plasma about 0.2 to 0.4%) while the blood sugar decreased. These all presented a history of infection previous to the onset of the diabetes and suffered from a more severe form of the disease than those of the preceding group. I. G.

Clinical calorimetry. XIX. The metabolism in tuberculosis. WILLIAM S. McCANN AND DAVID P. BARR. Russell Sage Inst. and Bellevue Hosp., New York. *Arch. Intern. Med.* 26, 663-705(1920).—The basal metabolism of tuberculous patients may be normal or very slightly above that of normal men of the same size; in 10 cases, the variation was from -3 to +15%. Rise in temp. increases metabolism; a rise of 1° by only 2 cal. per hr., but a rise to 40° may increase metabolism by 30%. The basal metabolism may be less than in the same individual in health because the lowering of metabolism due to the loss in wt. may more than compensate for any increase due to the disease. In 4 expts. the N excretion was reduced to 5 or 6 g. per day (about 0.1 g. per kg.) but equil. could not be attained below 10 g. "The specific dynamic rise produced in 2 cases by the ingestion of a protein meal corresponded closely with that produced by the same meal in 3 normal men. . . . In view of the fact that the food requirements of tuberculous patients are not larger, either as regards total energy value or nitrogen content, forced feeding is unnecessary and is probably harmful in the active stages of pulmonary disease. Since protein increases the respiratory exchange in the tuberculous as in normals it may be well to limit the protein intake during periods of activity (of the disease) in order to put the lungs at rest." I. GREENWALD

KOLTSCH, R.: Die Reiztheorie und die modernen Behandlungsmethoden des Diabetes. Berlin und Vienna: Urban und Schwarzenberg. 152 pp. For review see *Endocrinology* 4, 607(1920).

LACOG, R.: Les nouvelles théories alimentaires. Besoins en calories. Maladies par carence. Valeur des protéines. Vitamines et antavitaminoses. Importance des sels

minéraux. Aliments et alimentation. Paris: Vigot Frères, 23, rue de l'École de Médecine. 6 fr. For review see *Répert. pharm.* 32, 848(1920).

F—PHYSIOLOGY

ANDREW HUNTER

Hemato-respiratory functions. VII. The reversible alterations of the carbonic acid: sodium bicarbonate equilibrium in blood and plasma under variations in carbon dioxide tension and their mechanism. HOWARD W. HAGGARD and YANDELL HENDERSON. Yale Univ. *J. Biol. Chem.* 45, 189-98(1920); cf. *C. A.* 14, 3710.—VIII. The degree of saturation of the corpuscles with hydrochloric acid as a condition underlying the amount of alkali called into use in the plasma. *Ibid* 199-207.—IX. An irreversible alteration of the carbonic acid: sodium bicarbonate equilibrium of blood, induced by temporary exposure to a low tension of carbon dioxide. *Ibid* 209-14.—X. The variability of reciprocal action of oxygen and carbon dioxide in blood. *Ibid* 215-7.—XI. The relation of hemolysis to alteration of carbonic acid: sodium bicarbonate equilibrium. *Ibid* 219-21.—These papers are studies of the relation of the corpuscles to acid-base equil. in oxalated and in defibrinated dog blood. Bloods equilibrated with air containing no CO_2 contain no NaHCO_3 . As the CO_2 content of the equilibrating air increases, the NaHCO_3 of blood and plasma increases until in the expt. cited it reached a max. for plasma at about 61 vols. % between 72 and 146 mm. and for whole blood at about 80 vols. % between 360 and 440 mm. Corpuscles equilibrated with plasma at any tension of CO_2 will give off more alk. to plasma or NaCl soln. if washed with NaCl soln. and again treated with CO_2 . Even after equilibration at 712 mm. CO_2 , the cells yielded as much alkali (91 vols. % CO_2) to the NaCl soln. as they originally did to the plasma, both being measured at 42 mm. CO_2 . But this identity is a coincidence for corpuscles from blood initially equilibrated at 41 mm., then centrifuged and washed, yielded 104 vols. % when treated with NaCl soln. and 712 mm. CO_2 . "The total capacity of the corpuscles for acid is equivalent to an amt. of alkali several times greater than that in the plasma of normal blood. This capacity is controlled immediately by the tension of CO_2 and concn. of H_2CO_3 and presumably the H -ion concn. in the blood. When the H_2CO_3 is increased there is an almost proportionate increase of NaHCO_3 and the change in the CO_2 ratio ($\text{H}_2\text{CO}_3:\text{NaHCO}_3$) and H -ion concn. is correspondingly slight." In bloods that have not been exposed to abnormally high or low tensions of CO_2 , there is a nearly const. difference and this property may be used to judge of the "normality" of the blood. But below about 20 mm. CO_2 , the blood seems to undergo an irreversible, or not readily reversible change, after which it will, if equilibrated with a normal CO_2 tension, combine with less CO_2 than it did at this tension previously. Defibrinated blood is more apt to show such changes than is oxalated blood. Blood exposed to such low tensions of CO_2 no longer show the const. difference between CO_2 content of blood and plasma at different CO_2 tensions (or between the plasmas at different tensions and the bloods from which they were obtained) and are also very apt to show hemolysis. O in increasing concns. diminishes the CO_2 capacity of defibrinated blood but not, or not so regularly, in oxalated blood.

I. GREENWALD

Some variations in normal blood sugar. SOLOMON STROUSE. Nelson Morris Inst. Michael Reese Hosp., Chicago. *Arch. Intern. Med.* 26, 751-8(1920).—By means of purgation, by refraining from drinking or by drinking unusually large quantities (1 l. in 1 hr. before drawing the blood), an attempt was made to produce such changes in blood vol. as might affect the concn. of blood sugar. This was not successful. With the 5 subjects employed, it was found that the highest blood sugar values (0.11%) were obtained on clear cool days and the lowest (0.06%) on warm humid days.

I. GREENWALD

Creatinine and creatine in the blood. CHI CHE WANG AND MAMIE L. DENTLER. Univ. Chicago. *J. Biol. Chem.* 45, 237-43(1920).—By the methods of Folin and Wu (C. A. 13, 2541), creatinine (K_1) and creatine (K_2) detns. were made on the bloods of 24 women between the ages of 20 and 41. The values found were from 0.95 to 1.65, av. 1.30 mg. K_1 and from 2.23 to 4.65, av. 3.20 mg. K_2 , per 100 cc. There was no increase with age, nor was there any regular variation in the 15 cases in which blood was examd. during both menstrual and intermenstrual periods. Bloods taken 1 or 2 hrs. after a meal containing 70 g. sugar gave values for K_1 higher by from 0.07 to 0.39 mg., av. 0.20 mg., and for K_2 higher by an av. of 0.25 mg., than those taken after a normal meal.

I. GREENWALD

Blood phosphates in the lipemia produced by acute experimental anemia in rabbits. W. R. BLOOR. Univ. Calif. *J. Biol. Chem.* 45, 171-87(1920).—A study was made of the P compds. of the blood of rabbits subjected to repeated bleedings and in that way rendered anemic, with consequent lipemia. The greatest change observed was in the lipid P, which increased to over twice the normal in the corpuscles and to 5 times the normal in the plasma. When the lipid P was so greatly increased, the inorg. P of both plasma and corpuscles was also increased but there was no increase in the org. P of the corpuscles and only a variable increase in this fraction in the plasma. The only form of P which was notably higher in newly formed corpuscles than in older ones was the lipid P, but it is not certain whether this is characteristic of young corpuscles or is due to the lipemia.

I. GREENWALD

The physiological effects of short exposure to low pressure. E. S. SUNDBSTROM AND W. R. BLOOR. Univ. Calif. *J. Biol. Chem.* 45, 153-70(1920).—"From comparisons of the % changes in the number of erythrocytes, of the corpuscle vol., and of the P content of corpuscles and plasma of rabbits that have been subjected to a small hemorrhage (15-25 cc.) and then exposed for a short period (generally 3 hrs.) of time on the one hand to normal pressure and on the other hand to pressures from 350 to 450 mm. it is concluded that: (a) While at normal pressure the change of the number of red cells corresponds to the loss of blood from the exptl. hemorrhage, the greater alteration of the number of erythrocytes at lower pressures is due to the low pressure. (b) At very low pressures a decrease in the size of the red corpuscles occurs. (c) The P content of the red cells does not undergo any marked change. In a few cases the decrease of the lipid P is probably beyond exptl. error. (d) The change in the inorg. P of the plasma is negative, and of the same amplitude in control and low pressure series. (e) The most characteristic feature of the low pressure expts., when compared with the controls, is a const. decrease in the former of the lipid P of the plasma. It is suggested that the decrease of the lipid P of the plasma at low pressure indicates an enrichment of the erythropoietic organs with lipid material and that this may be the first phase of a stimulation of these organs resulting from a lowering of the atm. pressure."

I. GREENWALD

Contributions to the biochemistry of iodine. III. The comparative effects of thyroid and iodide feeding on growth in white rats and in rabbits. A. T. CAMERON AND J. CARMICHAEL. Univ. Manitoba. *J. Biol. Chem.* 45, 69-100(1920).—Continued small doses of desiccated thyroid fed to young white rats produce (a) a definite and invariable decrease in growth rate which is proportional to the amt. and I content of the thyroid fed; (b) a hypertrophy of organs concerned with increased metabolism—heart, liver, kidneys, adrenals, etc., which varies with dose and length of application and appears to be proportional to the I content of the thyroid fed; (c) an inhibition of growth rate of thyroid, and (d) a disappearance of fat. Similar results were obtained with young rabbits. No such effects were observed after the feeding of NaI in doses containing I in quantities varying from the equiv. of that fed as thyroid to amts. 100 times as great.

I. GREENWALD

GLAY, E.: Quatre leçons sur les sécrétions internes. Paris: J. B. Baillière. 154 pp. 7 fr. For review see *J. Am. Med. Assoc.* 75, 1444(1920).

G—PATHOLOGY

H. GIDEON WELLS

Sodium bicarbonate tolerance in the toxemias of pregnancy. CLIFFORD WHITE. London. *Lancet* 1920, II, 1248-9.—The av. bicarbonate tolerance of patients clinically suffering from pregnancy toxemia was found to be 45.3 g. compared with an av. of 6.7 g. in women not suffering from toxemia. A fatal case had a tolerance no less than 120 g., compared with an av. tolerance of 40 g. in those that recovered. The tolerance expresses the number of g. by mouth necessary to render the urine alk. R. B. FINK

Observations on the chemical pathology of the blood in pernicious anemia and other severe anemias. AMOS W. PETERS AND A. S. RUBNITZ. Univ. Nebraska Med. Coll. *Arch. Intern. Med.* 26, 561-9(1920).—The first 2 drops of finger blood are received in a weighed, stoppered test-tube containing exactly 12 cc. of a soln. containing 3.5 g. NaF and 5 g. NaCl per liter, made just alk. to phenolphthalein. After weighing, 2 portions of 2 cc. each are removed for detn. of total N (cf. *C. A.* 14, 752). The remainder of the mixt. is centrifuged and 5 cc. are taken for detn. of plasma N. For non-protein N, 10 drops finger blood are received in 10 cc. H₂O and treated as described in *C. A.* 14, 752. The technic is slightly modified in that the Nessler soln. is now prepd. by mixing 200 cc. of the Folin-Denis K₂HgI₄ soln. (*C. A.* 13, 2541) with 100 cc. 10% NaOH and 200 cc. H₂O. 10 cc. of this are measured into a 50 cc. vol. flask and the test soln., dild. to 40 cc., is delivered below the level of the Nessler soln. The total solids and the total N of the blood are lowered in pernicious anemia and in secondary anemias, the lowering being proportional to the decrease in the number of red cells. Plasma N is either normal or slightly above normal. I. GREENWALD

Observations on alimentary hyperglucemia. SOLOMON STROUSE. Michael Reese Hosp., Chicago. *Arch. Intern. Med.* 26, 759-67(1920).—In a normal person, following the ingestion of 100 g. glucose, the blood sugar rises to a max. of 0.15% and then falls. In a typical diabetic, the max. is higher and is not attained until after 1 hr. or longer. In a diabetic whose tolerance has been raised by dieting, a "normal" reaction may be obtained. Again, a "diabetic" reaction may be obtained in normal individuals if the amt. of glucose be increased. "Diabetic" reactions may be obtained in a number of pathological conditions, such as cirrhosis of the liver, hyperthyroidism, chronic arthritis etc., so that the reaction is not specific. "Nevertheless, alimentary hyperglycemia has a value in a differential diagnosis in obscure cases—a value entirely nonspecific and relative to other clinical and laboratory data obtained in the individual case." I. GREENWALD

Renal glucosuria. SOLOMON STROUSE. Michael Reese Hosp., Chicago. *Arch. Intern. Med.* 26, 768-74(1920).—A report on 4 cases of renal glucosuria, 2 under observation 2 yrs.; 1, 5 yrs., and 1, 8 yrs. There was no evidence of hyperglucemia except that in the 2 under observation longest, an alimentary hyperglucemia and a "diabetic" blood sugar curve (cf. preceding abstr.) were obtained. The patient under observation for 8 yrs., now 31 yrs. old, showed no depression of kidney function except in regard to excretion of phenolsulfonephthalein (23% in first hr. and 9% in second). I. GREENWALD

The failure of antibody formation in leucemia. KATHARINE M. HOWELL. Michael Reese Hosp., Chicago. *Arch. Intern. Med.* 26, 706-14(1920).—"Individuals with leucemia who contract typhoid or paratyphoid infection may fail to develop the sp. agglutinins in the blood." There is similar failure to develop agglutinins and opsonins after the injection of typhoid vaccine and the temp. and leucocyte reactions are variable and slight. I. GREENWALD

Relation of the alkali reserve of the blood to glucosuria and hyperglucemia in pancreatic diabetes. BYRON M. HENDRIX AND CAROLINE Y. CROUTER. Univ. Penna. *J. Biol. Chem.* 45, 51-5(1920).—Hyperglucemia and glucosuria appeared in pancreatectomized dogs before there was any decrease in the CO_2 capacity of the plasma. On the 1st or 2d day following the operation there was a slight increase in the CO_2 capacity which then fell gradually to as little as 30% on or about the sixth day. The results do not support Murlin and Sweet's theory (*C. A.* 11, 482) of the causal relation of the acid of the gastric juice to pancreatic diabetes.

I. GREENWALD

Relationship between the plasma bicarbonate and urinary acidity following the administration of sodium bicarbonate. WALTER W. PALMER, HARALD SALVESEN AND HENRY JACKSON, JR. Presbyterian Hosp. New York and Johns Hopkins Hosp. *J. Biol. Chem.* 45, 101-11(1920).—"In normal and pathological cases, following the administration of NaHCO_3 , the plasma HCO_3 approaches 68.7 ± 10 vols. % at the time the first depressing effect of the alk. on the urinary acidity is noted. The use of the first significant effect on the H-ion concn. of the urine, therefore, in controlling the therapeutic use of NaHCO_3 is safe and reliable. . . . The variability among the several levels of plasma HCO_3 at which a significant depression in the H-ion concn. of the urine occurs in normal and pathological cases is probably explained by the varying degrees of injury to the acid-base regulatory functions of the kidney. There appears to be no close relationship between this function and the ability of the kidney to excrete phenolsulfonephthalein or the blood urea." This same variability makes it impracticable to use the amt. of NaHCO_3 required to change the reaction of the urine as basis for calcg. the decrease in alk. reserve.

I. GREENWALD

New experimental research on the vaccination of cattle against tuberculosis. A. CALMETTE AND C. GUÉRIN. *Ann. inst. Pasteur* 34, 553-60(1920).—Heifers vaccinated with live bovine bacilli that had been cultivated on glycerol broth containing bile were resistant to injection with tuberculosis.

E. R. LONG

Studies on the mutual precipitation of antigens and antibodies. II. Antitoxic sera. M. NICOLLE, E. CÉSARI AND E. DEBAINS. *Ann. inst. Pasteur* 34, 596-9(1920); cf. *C. A.* 14, 3107.—A ppt. forms when solns. of antigens and antibodies are mixed. The strength of antitoxic serum can be detd. by mixing toxin with increasing dilns. of antitoxin by the method previously described. An antitoxin which in 1:50 diln. just gives a light cloud when overlaid on a standard soln. of toxin, has a concn. of 300 units per cc. in the case of diphtheria antitoxin, and 4000 in that of tetanus antitoxin, as learned by *in vivo* comparison. The standard toxin is prepd. by pptg. the original filtrate with Na_2SO_4 , taking up 0.8 g. of the dry powder in 10 cc. of water, and mixing this with equal pts. of 10% gelatin in physiol. salt soln. The mixt. is tubed in 1 cc. amts. and allowed to solidify. The method is a great saving of time, effort and money, as compared with the *in vivo* method of detn. of antitoxin strength.

E. R. LONG

Congenital steatorrhea. REGINALD MILLER AND HERBERT PERKINS. *Quart. J. Med.* 14, 1-9(1920).—An example of congenital steatorrhea is reported. The patient had passed liquid fat since birth and continued to do so even on a diet remarkably low in fat. This failure of fat absorption did not result in definite stunting, by the age of 3 years. There were no other definite pancreatic symptoms. The urinary diastase was low. There was no microscopic evidence of failure in protein digestion. There was probably an "inborn error" of metabolism, probably a lack of an enzyme, possibly a pancreatic enzyme.

JOHN T. MYERS

Some observations on the blood sugar in diabetes. DENNISON V. PICKERING. *Quart. J. Med.* 14, 19-56(1920).—The blood sugar values were carefully followed in 50 cases of diabetes. The amt. was almost invariably raised and it usually increased with the duration of the disease. It varies directly with the severity of the disease except that in mild cases with albuminuria or heavy consumption of alc. it is often

high. It is reduced by fasting. The reaction of blood sugar to treatment is more important than its height. A blood sugar which remains high in spite of treatment is not invariably a bad sign, but requires caution in increasing the carbohydrate diet. The amt. of blood sugar corresponds more closely to the intake in mild than in severe cases. A high renal threshold is not necessarily a bad sign. The amt. of carbohydrate in a meal a few hours previous to examn. does not affect the blood sugar. There is no striking relation between the renal threshold and the age of the patient or the duration of the case.

JOHN T. MYERS

The diagnosis of diseases of the pancreas with special reference to diastase in the urine. R. L. MACKENZIE WALLIS. *Quart. J. Med.* 14, 57-87(1920); cf. *C. A.* 14, 3271.—There is no one test yet devised which is pathognomonic of pancreatic insufficiency. The association of three tests, the increased diastase content of the urine, the Loewi adrenaline mydriasis test, and the presence of glucosuria, affords strong evidence that the pancreas is at fault. If there is in addition, creatorrhea and steatorrhea, the suspicion is confirmed. The conditions which produce these positive results are transitory. Certain tests, especially the estimation of trypsin and diastase in the oil test meal and feces, the Sahli test, Kashiwado's modification of Schmidt's test, and the Cammidge "pancreatic" reaction are of doubtful value.

JOHN T. MYERS

The effect of high protein diet on albuminuria and blood urea in cases of nephritis. R. WORDLEY. *Quart. J. Med.* 14, 88-101(1920).—Variations in the amt. of protein in the diet appear to have no effect on the protein excreted in the urine. If only percentages are taken the albuminuria might appear to decrease because of the diuresis caused by the rich protein diet. The amt. of protein in urine examd. at different times of the day shows wide variations. It seems to be lowest in urine excreted during sleep. Blood urea varies considerably with the diet. An estimation of blood urea is of less value than Maclean's modification of Van Slyke's method. The blood urea may be within normal limits and the manifestations of uremia present. The plasma protein may be below normal in cases with no edema and thus cannot be the cause of the edema.

JOHN T. MYERS

The kinetics of inactivation of complement by light. S. C. BROOKS. Harvard Univ. *J. Gen. Physiology* 3, 169-83(1920).—The apparent course of the photo-inactivation of complement by ultraviolet light is that of a monomolecular reaction. Diffusion is not the limiting factor because its temp. coeff. is higher than that of photo-inactivation. No change in transparency of serum solns. took place during photo-inactivation with light of about 2530 Å. Under these conditions, it is believed only a single disappearing molecular species governs the rate of reaction during photo-inactivation. This substance must be primarily responsible for the hemolytic power of serum when it is used as complement. The mechanism of complement action. *Ibid* 185-201.—When complement is exposed to ultraviolet light it is not sensitized to the action of heat which indicates that it is not a protein. Inactivation is accompanied by a decrease in the surface tension of complement; it is not the result of any changes in H-ion concn. H-ion concns. high enough to transform serum proteins from cation to anion condition permanently inactivate complement. The hypothesis is advanced that a hemolytic substance is present in serum which is formed from a precursor and is constantly being formed and broken down into inactive products. It may resemble lecithin. Both precursor and lysin contain the photosensitive molecular groups. The lytic substance is dependent for its activity upon the state of the serum proteins.

CHAS. H. RICHARDSON

Potency of some old human isohemagglutinating serums. M. W. LYON, JR. *J. Am. Med. Assoc.* 75, 1002-3(1920).—Results showed that a sample of known parasthenic, II, and another of antiparasthenic, III, isohemagglutinating serum had kept preserved with 0.5% phenol in the icebox for one year. Their agglutinating activities

were good and they did not give nonsp. agglutinations. The same serums kept in a dry pulverized state in the ice box, and redissolved in water containing 0.5% phenol before testing, for the most part showed good agglutinating properties, but inferior to the phenol-preserved serums. They also showed a tendency to nonsp. agglutinations.

L. W. RIGGS

The role of calcium in tetany. ROBERT H. KUMMER. Univ. Geneva. *Presse medicale* 28, 765-6(1920).—The appearance of tetany following the hypofunctioning of the parathyroids, which regulate the Ca equil., the metabolism of guanidine, or more probably both, is due to the disturbance of the balance between the Ca and products of metabolism. The deficit of Ca deprives the cells of their defense, facilitating the entrance of toxic products of metabolism which give rise to tetany. H. A. SHONLE

H—PHARMACOLOGY

ALFRED N. RICHARDS

The influence of intravenous injection of hypertonic solutions upon the composition and coagulability of the blood. PAUL SCHENK. Breslau. *Z. exp. Med.* 11, 166-77(1920).—Infusions of hypertonic NaCl, glucose and Ca solns. are generally harmless. NaCl infusions up to 130 cc. 10% soln. aside from producing marked thirst and slight fever are without marked effect and produce a marked histogenic hydremia as a result of which there is an increase in coagulability of the blood. The hydremia lasts about 4 hrs. Such infusions may be used in hemorrhage following vascular rupture and stasis, particularly pulmonary edema. Infusions of glucose produce a similar effect. In this case the amt. available is about 200 cc. of a 10-20% soln. The latter is apparently too concd. and produces marked fever. Sugar fever as well as salt fever occurs during the summer months as well as at other times of the year. Ca solns. can be injected intravenously in greater concns. and in larger quantities than was hitherto supposed. The effect on the heart as indicated by electrocardiograms is not marked.

E. B. FINK

The source of the uric acid excreted in the urine after atophan. GEORGE GRAHAM. *Quart. J. Med.* 14, 10-18(1920).—The extra uric acid excreted in the urine after atophan cannot come from the blood as the total amt. in the blood is too small. The evidence that the uric acid deposited in the tissues can be redissolved is very weak.

JOHN T. MYERS

The role of alkali salts in edemas. LEON BLUM. *Presse medicale* 28, 685-8 (1920).—In certain cases of nephritis KCl has a diuretic action which surpasses that of other diuretics. In moderate doses KCl will increase the action of other diuretics. In case of cardiac weakness caution must be used in giving KCl. H. A. SHONLE

Chemotherapy. A. RICHAUD. *Presse medicale* 28, 613-7(1920).—Org. arsenicals are generally discussed. It is probable that other metals besides As when placed in suitable org. compds. will show increased anti-syphilitic action and diminished toxicity.

H. A. SHONLE

I—ZOOLOGY

R. A. GORTNER

The action of inhibitory nerves on carbon dioxide production in the heart of *Limulus*. WALTER F. GARREY. Tulane Univ. and Marine Biol. Lab. Woods Hole, Mass. *J. Gen. Physiology* 3, 163-8(1920); cf. *C. A.* 14, 3682.—Stimulation of the inhibitory nerves of the neurogenic heart of *Limulus polyphemus*, which corresponds to the vagus nerves of the vertebrate heart, results in a marked diminution of CO₂ production in the heart ganglion. On the other hand, stimulation of the ganglion which leads to increased activity of the heart results in an increased production of CO₂ by the ganglion. These expts. indicate "that inhibition of the automaticity of this ganglion by the action of its inhibitory nerves consists, not in a process of blocking,

but in a diminution of those chem. reactions in the ganglion cells which give rise to the production of CO_2 ."

CHAS. H. RICHARDSON

Radioactivity and physiological action of potassium. ROBERT F. LOEB. Rockefeller Inst. *J. Gen. Physiology* 3, 229-36(1920).—Eggs of the sea urchin, *Arbacia*, were used in these expts. from which the following conclusions were deduced: "The non-radioactive Cs ion can replace the K ion almost quantitatively in solns. required for the development of the egg of the sea urchin into swimming blastulae. Thorium chloride and uranium acetate (both salts of radioactive elements) cannot replace the KCl in the solns. required for the development of the egg. Thorium chloride and uranium acetate do not antagonize the action of the K contained in sea water upon the development of eggs." A critical discussion of Zwaardemaker's idea of the effect of K on cardiac action is given. Cf. *J. Physiol.* 53, 273(1919-20).

C. H. R.

Chemical character and physiological action of the potassium ion. JACQUES LOEB. Rockefeller Inst. *J. Gen. Physiology* 3, 237-45(1920).—Expts. on the eggs of the fish, *Fundulus*, showed that in cases of antagonism, the NH_4 ion acts more like K ion than Na ion which is in keeping with the closer resemblance in chem. behavior of NH_4 to K than to Na. The tolerance of eggs of the sea urchin, *Arbacia*, toward Li ion can be greatly increased (500% or more) if a certain amount of Na ion is replaced by K, Rh, or Cs ions. Although Na occupies a place in the periodic table between K and Li, the latter ions appear to deviate in their physiol. action in the opposite direction from Na ion. It is concluded that the behavior of the K ion in antagonistic salt action is due to its purely chem. character, i. e., its position in the periodic table or rather its atomic number and not to those activities which give rise to a trace of radioactivity. Cf. Loeb (preceding abstract).

CHAS. H. RICHARDSON

Environmental factors other than temperature affecting facet number in the banded mutant of *Drosophila*. JOSEPH KRAFKA, JR. Univ. Ill. *J. Gen. Physiology* 3, 207-10(1920).—*Drosophila* reared on pure Fleischmann's yeast in paste form matured earlier and showed an appreciable difference in the facet counts than individuals reared on the usual fermented banana medium. No significant difference was noted in the facet number between flies which matured in air of 35% and 60% humidity. Direct evapn. in a current of air of 60% humidity increased the mean facet number. In spite of these differences, however, K. believes that when a consistent procedure is followed and plenty of good moist food is present, the environment is practically constant except for variations in temp. and need not be taken into consideration in interpreting most of the breeding data now available for *Drosophila*. Cf. C. A. 14, 2385.

CHAS. H. RICHARDSON

12—FOODS

W. D. BIGLOW AND A. E. STEVENSON

Simple methods for the examination of acid phosphates for use in baking powder. FR. SSELIG. Hanover. *Z. Nahr. Genussm.* 40, 206-7(1920).—The methods are designed to give information on the acidity of unfamiliar forms of phosphates now found on the market. (a) "Bi-phosphate acid." Five g. phosphate + 25 cc. H_2O + 40 cc. of 40% $\text{CaCl}_2 \cdot \text{H}_2\text{O}$ are titrated without previous filtration with 0.5 N NaOH, using 10 drops of 1% phenolphthalein, to a permanent red. The "bi-phosphate acid value" = cc. used $\times 100$ = cc. N acid for 100 g. phosphate and depends on the content of $\text{CaH}_4(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$. (b) Monophosphate acid. The titrated mixt. from (a) is heated with 10 cc. 0.5 N HCl, cooled and titrated back to red with 0.5 N NaOH. The "monophosphate acid value" depending on the $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ present = (titrated cc. — 10) $\times 100$ = cc. N acid for 100 g. phosphate. It is urged that the dealers in

baking powder ingredients be required to guarantee a certain phosphate acid value on each lot of phosphate.

L. D. ELLIOTT

Investigation of the estimation of crude fiber. OTTO NOLTE. *Landw. Versuchsst.* 96, 325-37(1920); cf. C. A. 14, 1859.—N. studied the various factors which influence the result of a crude fiber estn., as size of vessel, manner of heating, purity and strength of chemicals, and method used. In comparing the Cross-Bevan, Weender, Mach, and Kalning methods, the first always gave a higher percentage and the last a lower percentage. Little difference was found in the results obtained by the Weender and Mach methods. The high results as obtained by the Cross-Bevan method indicate that some of the cellulose is dissolved in the Weender and Mach processes. It made no material difference whether or not the fats were extd. before the estn. of the crude fiber. Extns. made of hay showed that $(C_2H_5)_2O$, $(CH_3)_2CO$ and $CHCl_3$ were about equally effective. Material, the greater percentage of which passed a 1 mm. sieve, showed about the same fiber content as meal which did not pass a 1 mm. sieve.

F. M. SCHWARTZ

Influence of feed upon the fat content of milk. III. A. BEHRER. *Chemnitz. Z. Nahr. Genussm.* 40, 202-3(1920); cf. C. A. 14, 302.—The results of 20 stall tests during 1919 gave an av. fat content of 3.23% compared with 2.98% for 1918, while the av. yield per cow increased from 4.5 l. in 1918 to 5.2 l. in 1919. The view that the lack of feed during the war had no adverse effect upon the fat content is substantiated.

L. D. ELLIOTT

Influence of feed deficiency upon the composition of milk. J. STERN. *Kreuznach. Z. Nahr. Genussm.* 40, 204-5(1920).—A study of tests made on market milk and stall tests for the years 1910 to 1919 show that the deficiency of fodder during the war did not affect the fat content of milk, but did reduce the yield of milk. (Cf. preceding abstract.)

L. D. ELLIOTT

Physiological properties of goat milk. C. P. DE LANGLE. *Pacific Dairy Review* 24, No. 46, 18-9(1920).—Part I. Diacritical review of the properties and compn. of goat milk.

H. F. Z.

New method of testing cheeses. A. GEAKE. *J. Agr. Sci.* 10, 86-9(1920).—The degree of ripeness of a cheese was estd. from the extent of proteolysis as measured by the amino N. Sørensen's method of detg. amino N was found suitable. Grind 8 g. of cheese 3 times with 30 cc. of acetone and allow the residue to dry in the air. Shake 3 g. of this residue with 50 cc. of 0.1 N KOH for 1 hr. and filter. Titrate 20 cc. of the filtrate to neutrality with 0.1 N H_2SO_4 , using phenolphthalein as an indicator. Add 10 cc. of neutral HCHO soln. and titrate back as usual. Det. the total N in 5 cc. of the cheese soln. by Kjeldahl's method. The result is given as percentage of amino N in total N. The method was tested on various cheeses of known quality.

R. B. DEEMER

History of vegetable oils. DAVID WESSON. *Cotton Oil Press* 4, No. 7, 53-4 (Nov. 1920).—An address before the Institute of Independent Margarine Mfrs. *Margarine*, even though it does not contain vitamins, may be used with impunity by those living on the usual mixed diet. The desirable qualities in butter substitutes are good flavor, proper consistency and satisfactory keeping properties. It is difficult to obtain a margarine of the consistency of butter, but hydrogenated coconut oil has done much to solve this problem.

H. S. BAILEY

An automatic apparatus for the determination of moisture in cereals. MARCEL CAOPIN. *Compt. rend.* 171, 860-2(1920).—An app. is described and illustrated wherein the cereal is automatically introduced into a cylinder heated by elec. coils and the moisture liberated measured after condensation.

H. A. LEPPER

Finely ground flour and bread prepared therefrom. A. HEIDUSCHKA AND J. DÄNNIGER. *Würzburg. Z. Nahr. Genussm.* 40, 161-91(1920).—This is mainly a

study of the H_2O -sol. constituents in war flours, doughs, and breads with a preliminary study of suitable chem. methods for examg. such products. In the H_2O -sol. matter of flours were found maltose, sucrose, vegetable gums, and erythro- and amyloextrin. Solids, ash constituents, P- and N-containing substances and acidity were detd. upon flours, doughs, and breads, and upon the H_2O -sol. matter in each. The most completely ground flours yielded the largest H_2O -sol. material. The latter was decreased in bread by the use of hot H_2O in making the dough, while cold H_2O had the opposite effect. The larger the ash and N content of a meal the lower was the H_2O -sol. material. The addition of substitutes such as potato meal, did not affect the % of H_2O -sol. material. Enzymatic action caused increases in carbohydrates and P and N compds. The results indicated that leavened bread contains more H_2O -sol. material than baking powder bread or yeast bread. A modification of the *detn. of acidity* was evolved to avoid slow filtrations and obscured end-points. Twenty g. of bread is rubbed up with H_2O and made up to 200 cc. in a measuring flask, the mixt. allowed to stand 2 hrs. with occasional thorough shaking. After further shaking it is allowed to settle for 10 min., then 50 cc. of the supernatant liquid is pipetted off for titration. L. D. ELLIOTT

The determination of the nutritive value of potatoes. OTTO H. MATZDORFF AND EMIL O. GROSSGEBAUER. *Pharm. Zentralhalle* 61, 598-600(1920); cf. Reichard, C. A. 14, 1168.—The *detn.* of the nutritive value of the potato from the starch content calcd. from the sp. gr. is too variable. The difference between the starch content and the total solids is a const. (5.752%). The potatoes are dried at 50° and then to a const. wt. at 105°. The % dry wt. less 5.8% gives the starch value. H. A. SHONLE

Two peculiar food substitutes. JOHANNES PRESCHER AND REINHOLD CLAUS. *Cleve. Z. Nahr. Genussm.* 40, 208(1920).—In a valueless *substitute for abundance* no trace of essential oil of cinnamon was found. A *rice starch substitute* consisted of potato starch, 20% NaCl, and 35% foreign matter consisting of sand, clay, chalk, magnesia, and alkalies. L. D. ELLIOTT

New contribution to lactic silos. C. GORINI. *Rend. reale ist. Lombardo* 53, 1-8(1920).—Lab. expts. in which silage was made in glass jars with and without inoculation with lactic acid bacteria demonstrate the advantage of inoculation. The following figures represent the percent of non-volatile acid after 15 days incubation at 30-35°. Raw fodder, not inoculated, 0.432, inoculated, 0.493; sterilized fodder, not inoculated, 0.220, inoculated, 0.480. A. W. DOX

Studies on lactic silos from the standpoint of bacterial physiology. C. GORINI. *Rend. reale ist. Lombardo* 52, 192-295(1919).—In the preservation of forage by ensiling as practiced in Italy, an undesirable butyric fermentation is apt to occur unless special precautions are taken. If lactic acid bacteria are not already present in abundance they must be added by inoculation. Green fodder should be partially dried before ensiling. The addition of 1.5% NaCl and the avoidance of temps. above 30° favor the development of lactic acid bacteria and inhibit butyric fermentation. Milk from cows receiving butyric silage is unsuitable for making the milder varieties of cheese and should be used only for the strong, salty cheeses. A. W. DOX

Analysis by simple and reliable methods of the degree of decomposition of straw fodder. WISSEL. *Landw. Versuchsst.* 96, 256-77(1920).—The following methods along with other were tested: Phloroglucinol test, Weender method, gravimetric method, volumetric method after Stock and the chloride of lime method (Wohl). Origin of the samples tested is given. The results, as obtained by several analysts, are given comparatively in tables. F. M. SCHERTZ

Study of some poultry feed mixtures with reference to their potential acidity and their potential alkalinity. B. F. KAUFF AND J. E. IVRY. *North Carolina Agr. Expt. Sta. J. Agr. Research* 20, 141-9(1920).—Analyses were made of the mineral content of a number of poultry feeds with a view to detg. the potential acidity and potential

alkalinity of these feeds. The results show that the grain mixts. as ordinarily used in poultry feeding are acid. Mash mixts. containing sufficient quantities of digester tankage, meat and bone meal, dried milk or dried blood will be base. Acid balances of feed mixts. can be overcome by the addition of one or more of these base feeds; green feed, milk to drink, and limestone and oyster-shell grit also aid in overcoming the acid balance of grain mixts. Further studies are being made to det. whether acid feeds will in any way interfere with either growth or egg production. W. H. ROSS

The cultivation of "ghessab" (*Pennisetum spicatum*) at Rome. E. PANTANELLI. *Stax. sper. agrar. ital.* 53, 47-66(1920).—The value of *P. spicatum* as forage is known throughout northern and eastern Africa as well as in British India. Plants grown at Rome and gathered when mostly in flower contained 71.75% H₂O, 1.306% reducing sugars, 0.0303% sucrose and 1.65% starch. A portion dried at 90°, ground and exposed to the air contained 12% H₂O, 12.11% crude protein (of which 52.3% was digestible), 3.91% crude fat, 25.10% carbohydrates convertible to sugar, 36.14% cellulose, 10.16% ash, 1.937% total N, 12.803% hemicellulose and 1.47% total P₂O₅. Manus (*Dept. of Agr. Bombay Bull.* 77(1916)) found for "bajri" (caled. to 12% H₂O basis) 0.88% ether ext., 2.86% crude protein, 44% digestible carbohydrate, 34.76% fiber and 5.50% ash. The differences are ascribed to the latter sample having been taken from plants gathered during the rainy season while the former was made on plants which had come to flower after the longer dry summer. Hay from *P. spicatum* analyzed by Honcamp *et al.* (*C. A.* 7, 200) contained 12.91% H₂O, 14.67% crude protein, 13.66 pure albumin, 12.09% digestible protein, 4.40% ether ext., 76.18% non-nitrogenous ext. matter, 2.37% crude cellulose, 2.38% ash. ALBERT R. MERZ

Wild flax cake and fat-extracted mustard residues, their composition and their digestibility. F. HONCAMP, H. ZIMMERMANN AND O. NOLTE. *Landw. Versuchsst.* 96, 339-52(1920).—*Camelina sativa* Crantz cake had the following compn.: H₂O 11.77%, N-containing substances 33.10%, fat 9.21%, N-free extractive 27.65%, crude fiber 11.59% and ash 6.68%. Tests with sheep showed the digestibility of org. substances to be 65.5%, of crude protein 71.8%, of N-free extractive 64.7%, of ether ext. 32.5% and of crude fiber 66.3%. The cake contained 27.15% of digestible protein. Mustard cake contains about 35% of digestible protein, while meadow hay contains 6.65%. F. M. SCHERTZ

Cottonseed meal as a feed for hogs. J. C. BURNS. *Cotton Oil Press* 4, No. 7, 56-8(Nov. 1920).—Details of feeding expts. at the Texas Expt. Sta. with hogs using cottonseed meal in different proportions in the ration are given. Mixed with milo chops, the meal was fed with and without fermenting the mash. One group of hogs received also copperas in its ration. The results indicate that there is no advantage in fermenting the feed and that FeSO₄·7H₂O is of doubtful value. The greatest gain in wt. per head was made on one part cottonseed meal to 3 parts milo meal and the cost, per lb. gained, was lowest with this ration. If not over 10% of the whole ration is cottonseed meal it may be fed to advantage over long periods and without danger of injurious effects. H. S. BAILEY

Variations in the composition of wheat (LINDER) 11D. Respiration of cereal plants and grains (BAILEY, GURJAR) 11D. Occurrence of diastase in the sweet potato in relation to the preparation of sweet-potato sirup (GORE) 11D. The influence of various chemical and physical agencies upon *Bacillus botulinus* and its spores (WYANT, NORMINGTON) 11C. Bacterial inhibition. I. Germicidal action in milk (CHAMBERS) 11C. Specific-gravity tester (U. S. 1,360,537) 1.

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Pasteurization of Milk. Rept. of Comm. on Milk Supply of the Sanitary Engineering Section, A. P. H. A. Boston: A. P. H. A. 32 pp. 35 cts. For review see *Am. J. Public Health* 10, 985(1920).

Standard Methods for the Sanitary Analysis of Milk. 3rd Ed. revised. Boston: Am. Public Health Assoc. 30 cts. For review see *Am. J. Public Health* 10, 986(1920).

Food for animals. E. BECKMANN and VEREDELUNGSGES. FÜR NAHRUNGS-UND FUTTER MITTEL. Brit. 151,229, Aug. 15, 1919. Straw for fodder is softened by treatment with soda, potash, or other alkali lye, without artificial heat. Lyes of different concn. may be used successively, or previously used liquor may be followed by a fresh soln. To control the treatment, a removable gauze container may be employed inclosing knots which are examined periodically. Residual lye is removed by washing, a layer or filter paper, etc., being placed above the straw and a drainage orifice provided beneath. The product may be stored under pressure in silos, or dried.

13—GENERAL INDUSTRIAL CHEMISTRY AND CHEMICAL ENGINEERING

HARLAN S. MINER

Science and industry. F. HABER. *Chem.-Ztg.* 44, 913-4(1920). E. H.
Cost accounting in chemical industry. M. DOLCH. *Chem.-Ztg.* 44, 926-8, 953-4 (1920). E. H.

The chemical industry of Switzerland. A. LANDOLL. *Chem. Met. Eng.* 24, 21-2(1921). E. J. C.

Chemical disarmament. V. LEFEBURE. *Chem. Met. Eng.* 24, 5-7(1921).—"Chem. disarmament is the crux of all disarmament." An internationally legalized redistribution of org. chemical-producing capacity throughout the world is the method suggested. E. J. C.

The congress for industrial rights protection. Berlin, Oct. 21-3, 1920. FARTIG.. *Z. angew. Chem.* 33, I, 309-10(1920). E. J. C.

The engineering profession and government technology. R. S. McBRIDE. *Chem. Met. Eng.* 23, 1265-7(1920). E. J. C.

Lubricants and lubrication. ANON. *Chem. Trade J.* 67, 760-70(1920).—The report of the Lubricants and Lubrication Inquiry Committee issued by the Advisory Council of the Department of Scientific and Industrial Research is reviewed and a list of 13 recommendations for future research is given. EUGENE C. BINGHAM

Centrifugal extractors and separators applied to the chemical industry. F. J. BROADBENT. *Chem. Age (London)* 3, 652-7(1920); *Chem. Trade J.* 67, 772-3(1920); *Gas J.* 152, 638(1920); illus.—The problem of drying $(NH_4)_2SO_4$ is discussed. If the salt is acid, the cage should be made of acid-resisting material; Cu is probably the best. Since dried sulfate is a very fine powder, the perforations in the cage must be very small or a Cu-gauze lining must be used. Again, the outer case must be acid-proof (for this purpose a lead lining is usually used) and it must also be strong (of steel). The machine is loaded from the top and discharged through openings in the bottom.

of the cage and pan. These bottom-discharge extractors should preferably be of the suspended or Weston type rather than the under-driven type to facilitate operation and accelerate discharge. If the salt is neutral, a black steel cage should be used with a lead-lined pan. In all cases, where the temp. does not rise above 90°, a satisfactory coating for the cage is vulcanite. It must be continuous in structure and tested periodically electrically so as to avoid all chances of a leak of acid to the iron foundation and to prevent accidents. In actual practice a 48-inch cage with 7 charges in 2 hrs., each of 3 cwt., lasted 5 yrs. and the gauze 6 months. The extn. was carried out by rotating at 900 r. p. m. for 2 min. The moisture in the finished product amounted to just over 2% and the NH_3 to 25%. Two men were required for operating the machine and 4 h. p. was sufficient for driving. The drying effect is less with large-sized machines for practicable speeds than with small machines. The Weston, Broadbent, Gee and Sharples types are described.

J. L. WILBY

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Treating liquids with gases. H. R. HANLEY. U. S. 1,360,524, Nov. 30. In treating liquids with gases, e. g., in treating metal salt solns. with H_2S , the gas is introduced into a body of the liquid at a level above its bottom and the spent gas is then introduced into the body of liquid at a lower level than the point of first entry. This mode of operation serves to secure even treatment of the liquid with the gas.

Concentrating liquids. F. MERZ. Brit. 150,786, June 4, 1919. Air or other gas is caused to circulate in a closed cycle through two chambers, one containing the liquid to be concd. and the other hygroscopic material for drying the gas. The hygroscopic material is regenerated by passing air through it. A suitable construction is specified.

Filtering liquids. O. BAUMGARTNER. Brit. 151,620, Sept. 23, 1920. A filtering medium, such as finely granulated cork which will float in the liquid to be treated, is used. The cork is heated before use and is placed in the filter underneath a sieve

against which it rises, the liquid filtering upwards. It may be cleansed by the partial emptying of the space below. Application to the filtration of H_2O and of waste waters from paper manuf. is mentioned. For the removal of Fe from H_2O the medium may be "soaked in SnO_2 ."

Distilling and evaporating liquids. F. MERZ. Brit. 150,785, June 4, 1919. In evapg. liquids and recovering the vapors or gases obtained thereby, by means of a current of gas which circulates through a closed circuit comprizing an evaporator and means for sepg. the vapors from the gas, the evaporator comprizes stationary or moving surfaces which are alternately immersed in the liquid and exposed to the gas. The vapor-sepg. means may be either a surface or jet condenser or a hygroscopic or other absorbing liquid. In distg. alc., the vapors are condensed by contact with cold alc. In distg. NH_3 , H_2O is used as an absorbent. The app. may also be used for distg. hydrocarbons, and for concg. acids and sugar and saline solns.

Evaporating liquids to dryness. T. BOBERG and TECHNO-CHEMICAL LABORATORIES, LTD. Brit. 150,068, May 27, 1919. In evapg. liquids to dryness by process such as those described in 12,462, 1911 (C. A. 6, 3039), 22,670, 1911 (C. A. 7, 1119), and 149,055, in which the vapor generated is utilized, after compression, in heating the substance treated, the evapu. takes place in two stages, the liquid being first spread on a heated surface from which, when partly dried, it is removed by scrapers or otherwise, being then spread on to another surface. A suitable construction is specified.

Separating constituents of air or other gaseous mixtures in a rectifying column. R. WUCHERER and F. POLLITZER. U. S. 1,300,853, Nov. 30.

Bearing-fitting composition. H. C. HAGMAIER. U. S. 1,361,719, Dec. 7. A compn. for fitting hearings is formed of silica 22.25, white lead 22.25, graphite 44.5, and Na_2CO_3 11%.

Lubricant. H. F. WHELOCK. U. S. 1,362,161, Dec. 14. A pasty lubricant adapted for use on brake hands or drums is formed of kieselguhr or like material satd. with oil. Oil-bearing filtration cake may be used in prepg. the material.

14—WATER, SEWAGE AND SANITATION

EDWARD BARTOW

Water purification for boiler feed. F. J. CORLIUS. *Power Plant Engr.* 24, 1063-1121(1920).—A full discussion of the usual properties of characteristic combinations found in boiler feed waters is given. Permutite, lime-soda, and hot-process treatment are briefly described. Possibilities of compound treatment are mentioned. C. recommends frequent tests of water from the boilers to det. the internal conditions in properly adjusting whatever treatment is applied.

R. C. BARDWELL

The combined type sewage settling tank. W. S. COULTER. *Mun. County Eng.* 59, 192-4(1920).—The so-called combined tanks are discussed with special mention of splash devices for removing entrained gases. W. G. Kirchoffer urged sepn. of solids at the bottom of the sludge chamber and gas liberation.

LANGDON PEARSE

Surface shrinkage of rapid filter sand beds. WESTON GAVETT. *Eng. News-Record* 85, 1081-3(1920).—Hydraulics, design and operation are greater factors for surface shrinkage of sands than adsorption.

FRANK BACHMANN

Water-borne outbreak of typhoid at Schenectady, N. Y. THEODORE HORTON. *Eng. News-Record* 85, 1101(1920).—The water supply is obtained from 3 wells, one of which was polluted due to high water in the Mohawk River. The pollution was the cause of 53 cases and 3 deaths from typhoid.

FRANK BACHMANN

Sanitary disposal of sewage through a septic tank. H. R. CROHURST. *U. S. Pub. Health Repts.* 35, 2959-64(1920).—The data, cross-sections, plans, etc., presented

in this paper were prepd. in answer to frequent requests for a practical and economical method for sewage disposal for isolated homes or small groups of homes. This article is a description of a "free-flowing, tight, sewage tank" originally devised by Fletcher of the N. H. State Bd. Health. The tank is rectangular, built of concrete, without baffles, with a relatively tight cover, and without ventilation. Several figures showing plans and cross sections are given.

FRED W. TANNER

Notes on the design and principles of sewage siphons. WESTON GAVETT. *Eng. News-Record* 85, 1041-2(1920).

F. BACHMANN

Houston (Texas) activated-sludge plant results. J. C. McVRA. *Eng. News-Record* 85, 1128(1920).—Effluent was clear and inoffensive. Suspended solids were reduced 96 to 98% and relative stability varied from 86 to 98%. A reduction in O consumed of 50-81% was effected. Sewage required 1.44 cu. ft. air per gal. with detention period of 2 hrs. 15 min. Cost per million gals. was \$14 exclusive of overhead and sludge disposal. The 12,000,000-gal. plant was operated at $\frac{1}{2}$ its capacity. Cf. C. A. 14, 440.

FRANK BACHMANN

Activated sludge in England. EDIT. *Eng. News-Record* 85, 1018(1920).—Activated sludge plant for Reading, England, is approved by British Ministry of Health, to treat sewage of 100,000 persons. Sewage farms have been maintained for a number of years at a loss. American engineers should take note of the sanction by the Ministry of so large an installation of this process.

FRANK BACHMANN

Large activated-sludge installation at Reading, England. T. MIDGLEY TAYLOR. *Eng. News-Record* 85, 1125(1920).—The plant will comprize screens and grit chambers, 4 aeration tanks with total capacity of 2,100,000 gal., 2 settling tanks of 540,000 gal. total capacity, sludge-storage capacity 180,000 gal., and reaeration tank capacity of 312,000 gal. The dry-weather flow of sewage is 3,300,000.

FRANK BACHMANN

The Des Plaines River activated-sludge plant. LANGDON PEARSE. *Eng. News-Record* 85, 1134-8(1920).—The plant will consist of coarse rack screens, grit chambers, a Reinsch-Wurl screen, 4 aeration tanks and settling tanks, some of them provided with Dorr thickeners. The sewage of 4 towns with an aggregate population of 30,000 will be treated. The dry weather flow varies from 4 to 10 sec. ft. This plant will be a model plant with flexibility for expts. Sludge is to be filter-pressed and dried in a direct-indirect dryer. Dehydration of the sludge with a Besco ter-Meer centrifuge will be tried. Plans of the design are detailed.

FRANK BACHMANN

Report on Richmond Borough sewage tests, New York City. WARREN R. BORST. *Eng. News-Record* 85, 1130-1(1920).—Tanks with detention period from 1 to 2 hrs. and velocities of flow from 0.007 to 0.015 ft. per sec. removed 50-60% of suspended solids and 30-40% of oxidizable org. matter. Tank treatment followed by chlorination is recommended. In localities where covered tanks are necessary, the siphon tank is favored. Use of the waste steam and heat from the garbage destructors is recommended to dry the sludge, which can then be burned with the garbage.

FRANK BACHMANN

Garbage disposal in the District of Columbia. F. S. BESSON. *Eng. News-Record* 85, 1072-5(1920).—Garbage has been collected by the municipality since July 1, 1918. Collection covers 45 of the total land area of 60 sq. miles. About 53,000 tons of garbage were collected from 400,000 people. Disposal cost is \$4.15 per ton. Garbage is disposed of by the Chamberlain reduction process. Recommendations for expenditure of \$169,000 for the reduction plant and \$37,000 for transportation are made adequately to dispose of the garbage.

FRANK BACHMANN

Rubbish collection and disposal, District of Columbia. F. S. BESSON. *Eng. News-Record* 85, 1182-4(1920).—Street cleaning, collection and disposal of garbage, ashes and rubbish are conducted by municipal forces as private contracts were unsatisfactory. The rubbish is sorted for salable materials and tailings are burned.

Salvage pays 75% of cost of collection and disposal operating expenses. Salvage material consists of paper, rags, cans, metal, rubber, bottles, and broken glass.

FRANK BACHMANN

Studies on the corrosive action of chlorine-treated water (CLARK, ISELEY) 9.

KENWOOD, HENRY R.: Public Health Laboratory Work (Chemistry). 7th Ed. London: H. K. Lewis & Co. 420 pp. 15s. For review see *J. State Med.* 28, 387(1920).

Softening and purifying water. A. GAWALOWSKI and FIRMA JULIUS OVERHOFF. Ger. 319,436, July 17, 1914. Alkali, alk. earth or heavy metal ferrates or ferrites are employed as filter or contact materials or as additions to the H_2O under treatment. The ferrates or ferrites are obtained by fusing Fe with KNO_3 or $NaNO_3$. Hematite, magnesite, limonite, glauconite, goëthite, and weathered blast-furnace slags may be employed with the ferrates or ferrites. The latter are decomposed by the H_2O and the decompn. products, as formed, enter into reaction with the substances in the H_2O .

Removing iron from treated water. TH. GOLDSCHMIDT AKT.-GES. Ger. 319,963, Dec. 31, 1913. H_2O is filtered through sand. *E. g.*, conduit water is treated with chloride of lime soln. in an amt. to introduce about 7 mg. active Cl per l. The H_2O is then filtered, in a closed filter, over comminuted sheet iron. After a sample of the H_2O has been taken for analysis, the principal portion is filtered through a sand filter with exclusion of air. After filtration over Fe the H_2O contains 2.06 to 2.60 mg. Fe per l. After the filtration through the sand same H_2O contains never more than 0.2 mg. Fe per l.

Removing manganese from water. DEUTSCHE FILTERKOMPAGNIE, G. M. B. H. Ger. 319,605, Sept. 14, 1910. H_2O is conducted over trass or the like, which has been treated with Mn salts and then with permanganates. The higher Mn oxides which readily yield O ppt., by oxidation, the M^{++} compds. dissolved in the H_2O , thereby rendering them filterable. This filter may be regenerated by the passage of O, O_3 or substances yielding O.

Sizing; water-clarifying reagents. G. MUTH and L. DIVINAGE. Brit. 150,280, Aug. 17, 1920. Addition to 149,001 (C. A. 15, 295). In the process described in the principal patent, in which aluminiferous material is treated with a F compd. in the presence of H_2SO_4 , excess of acid is employed and this excess is neutralized with compds. of Mg or other alk. earth metal, or of Al. As neutralizing agents, $MgCO_3$, Mg, dolomite, $Al(OH)_3$, aluminates, and the product obtained when CaO is added to Mg sulfate soln., are mentioned. The mixts. obtained can be used for sizing paper or clarifying H_2O .

Water filter. W. MAPLE. U. S. 1,362,125, Dec. 14.

Treating sewage sludge or similar waste organic materials. A. MACLACHLAN. U. S. 1,360,427, Nov. 30. An oxidizing gas such as SO_3 is passed through a mass of sludge, garbage or similar material and the unspent gas is successively passed through other masses of similar material while oil is allowed to ppt. on the heavier oxidized substances present.

15—SOILS, FERTILIZERS AND AGRICULTURAL POISONS

J. J. SKINNER

Relations existing between the soil and its water content. B. A. KERN. *J. Agr. Sci.* 10, 44-71(1920); cf. C. A. 14, 1723.—This paper gives a résumé of investigations on moisture in soil and behavior under varying conditions. The max. H_2O capacity and pore space, permeability, capillary effects and H_2O -retaining capacity,

capillary movement of soil H_2O , hygroscopic moisture, and surface forces are the subjects treated. This last subject is considered from the point of view of the colloidal hypothesis, and it is considered that one of the most important soil constituents is the colloidal clay. Conclusions based on an examn. of soil soln. after it has been removed from the soil are open to doubt. It is essential that the soil and the soil soln., be treated as a whole.

R. B. DEEMER

The form of acidity of soils and its meaning in plant physiology. H. KAPPEN. *Landw. Versuchsst.* 96, 277-307(1920).—Three forms of acidity are recognized: (1) Acidity due to free acids and acid salts. (2) Treatment of soil with a soln. of pure neutral salt gives another form of acidity. (3) Acidity due chiefly to strong bases and weak acids, resulting from hydrolytic action. These forms of acidity are discussed at length as to their causes and effects upon plant growth. Particular attention is given to the effect of H ions. The 3d form of acidity is not harmful to plants. Soil acidity depends more upon the Ca poverty of acid soil than upon the latent properties of acidity resulting from hydrolysis. From the plant physiologist's viewpoint humus acids are not believed to possess any harmful action. Strong inorg. acids, especially free H_2SO_4 , are the ones believed to cause greatest injury. Cf. C. A. 14, 191.

F. M. SCHERTZ

Determination of ammonia in soil. D. J. MATTHEWS. *J. Agr. Sci.* 10, 72-85 (1920).—The app. used in the detn. of NH_3 in soils consisted of a long, wide aeration tube placed in a sloping position, the lower end being closed with a cork carrying the air inlet tube, and the upper end connected to the bottom of an absorbing app. consisting of an upright tube with 2 bulbs and with a splash retainer in its upper end. Twenty-five g. of soil is placed in the lower part of the aerator, and 50 cc. of soln. of Na_2CO_3 and chloride and 1 cc. of paraffin are added. 0.02 N H_2SO_4 and a few drops of methyl red are placed in the absorber, and a current of air washed through acid is drawn through the app. at the rate of 300 l. per hr. for 6 hrs. With most soils the efficiency of the app. was over 98%. With soils containing much undecompd. org. matter, it is better to replace the usual alk. soln. by a soln. of Mg in strong NaCl and to aerate for a fixed period of 3 hrs. Low results were always obtained with soils high in Ca unless the samples were very finely ground.

R. B. DEEMER

Absorption of phosphoric acid and soil reaction. MARGARETE WRANGELL. *Landw. Versuchsst.* 96, 209-55(1920).—Corn makes use of difficultly sol. phosphate only when aided by an acid reaction which may be brought about by fertilizers. Addition of lime neutralizes the acid reaction and causes the P_2O_5 absorption to cease. Mustard is able to absorb difficultly sol. P_2O_5 when the reaction is more alkaline. It is, therefore, independent of lime addition, alkaline or acid reaction, so long as the lime added does not produce a poisonous effect. Corn absorbs lime in the ratio of 3 mols. of CaO to 1 mol. of P_2O_5 . Mustard requires 15 mols. of lime for the same amt. of P_2O_5 . The process of making sol. the nutrient materials in the soil appears to depend on the alternate absorption of cations and anions, which is effected by alternate acid and alkaline reactions. As a rule one finds accelerated cation absorption at the beginning of vegetation, which produces favorable conditions for an increased absorption of P_2O_5 in the second stage of growth. Addition of basic or acid solns. accelerates the introduction of the nutrition process. N fertilizers are of especial value in nutrition and in regulating the soil reaction. By using N in the form of cation as NH_4 , and as an anion in the form of KNO_3 or in the form of NH_4NO_3 , optimum conditions for plant growth can be brought about. Thus free citric acid is more quickly decomposed in the soil. The presence of CO_2 in the soil clearly appears to play a role in the dissolving and decomp. process. Results in the paper make it easier to interpret the following phenomena: contradictory results of K fertilizing, soil fatigue and fruit growth, the K sensitiveness of some plants which have a proportionately high K requirement,

the origin of colloids through the alternate acid and alk. reaction within the working range of plant roots.

F. M. SCHERTZ

The occurrence of substances injurious to vegetation in certain volcanic ash deposits. J. TH. WHITZ. *Med. v. h. Lab. v. Agroeologie en Grondonderzoek v. h. Alg. Proefst. v. d. Landbouw*, No. 6, 1920; *Arch. Suikerind.* 28, 1891-901(1920).—The various layers of volcanic ash deposited through a volcanic eruption occurring in Java in 1920 were analyzed. Percolation of several ash samples with rain water showed that they contained less than 1% of water-sol. material, the finely divided ashes yielding more than the coarser ones. In the water exts. no injurious constituents could be detected, and corn plants grew normally in them. The water-insol. part of the ashes consisted of volcanic glass, pyroxene, amphibole, plagioclase, and ore. The samples from all but two localities contained no injurious substances. In the other two samples a bluish, and also a brown layer were detected which were rich in pyrites and pyrrhotite. These minerals upon weathering give rise to H_2SO_4 , which is injurious to plants. Under certain conditions H_2S may be formed, which is also poisonous. Where volcanic ash of this nature has been deposited, the soil must be carefully worked in such a way that the sulfides are rapidly oxidized, and the land must be well drained, to wash out the oxidation products as quickly as possible. Applications of lime are also recommended.

F. W. ZERBAN

Washing out of nitrates by drainage water from uncropped and unmanured land. E. J. RUSSELL AND E. H. RICHARDS. *J. Agr. Sci.* 10, 22-43(1920).—Data are given secured in the expt. with 3 natural soil gages in operation at Rothamsted since 1870. The soil was untreated, grew no crop, but was kept free from weeds. The loss of nitrate from the soil though continuous throughout the whole period of the expt. was remarkably slow, the N removed per annum being 40 lbs. per acre. The amt. of N removed at any time varied with the rainfall, 1 lb. of N per acre being removed by 1 in. of rain as an av. This may be accounted for by the fact that the NH_3 and nitrate formed as a result of the decompn. of the org. matter is possibly assimilated by organisms, such as algae and bacteria, and that only on the death of these is part of the N given out and part re-assimilated. The total Cl in the rain water during the whole period agreed with the total Cl in the drainage H_2O which is considered as evidence of the reliability of the results given.

R. B. DEEMER

The cause of nitrogen loss from liquid manure and from stable manure. OTTO NOLTE. *Landw. Versuchsst.* 96, 309-24(1920).—Volatility of NH_3 is given as the cause of N losses from urine. The loss of N is closely associated with CO_2 losses, the relation of N_2/CO_2 being 0.75 for urine. Evapn. of water is not the cause of loss of N_2 . The tension of NH_3 and CO_2 in urine is given as the sole basis of the rapidity of the loss. The effects of adding inorg. salts to liquid manure, for the purpose of retaining the NH_3 , are tabulated and each is discussed. Gypsum conserved about 70% of the NH_3 . The advisability of using humus coal for conserving NH_3 concludes the paper. Cf. C. A. 14, 1868.

F. M. SCHERTZ

Protein content of wheat. W. F. GERICK. *Univ. Cal. Science* 52, 446-7 (1920).—Pot expts. were carried out in triplicate in 2 series in one of which the N was furnished by $NaNO_3$ and in the other by $(NH_4)_2SO_4$. The soil used was poor and notably deficient in N. Fertilizer was added at the rate of 100 lbs. N per acre to the different sets of pots at the time of planting, 17, 33, 48, 72, and 110 days resp. after planting. The yield of grain of a pure strain of Australian wheat was 9.4, 10.6, 21, 19.9, 21.9 and 13.1 g. and the corresponding percentages of protein were 8.6, 9.3, 10.4, 11.8, 13.2, 15.2 resp. The data show an increase of 77% in the protein content of wheat obtained from plants that received N when they were 110 days old over those that received N at the time of planting. The com. grades of wheat changed from No. 2 soft in the first 2 sets of pots to No. 1 hard in the last 2. It appears that low protein

in Pacific States wheat is not due to climate but rather to the failure of the soil to furnish enough available N at the right period of growth. L. W. RIGGS.

The question of carbon dioxide fertilizing. HUGO FISCHER. *Fähling's Landw. Zig.* 69, 289-92(1920).—A critical review of the work of Gerlach (*Mitt. deut. Landw. Ges.* 27, 370-71(1920)) in Bromberg on the effect of CO_2 on the growth of tobacco, corn, tomatoes, etc. Cf. C. A. 14, 3291. F. M. SCHERTZ.

The role of copper in anti-cryptogamic sprays. VIALA. *Compt. rend. agr. France* 6, 754-6(1920).—Villedieu states that Cu does not possess its ascribed toxic properties and that it is without action on zoospores of mildew. The spores, he believes, are prevented from germination by the action of acid or base, in excess. Viala employed sodium biacetate against the mildew with good success, but the leaves retained the spray with difficulty. F. M. SCHERTZ.

Fungicidal dusts for control of smut. W. W. MACKIE AND FRED N. BRIGGS. *Science* 53, 540-1(1920).—Results compiled from repeated tests indicate that a mixt. of equal parts of CuSO_4 and CaCO_3 can be used more effectively in the control of smut when applied as a dust than when used in soln. CuCO_3 dust is equally effective. These dusts adhered tightly and completely covered all parts of the wheat seed used in making the tests, and no damage to seed germination occurred. Two ounces of the dusts per bushel are considered ample. W. H. ROSS.

The determination of cyanamide nitrogen in commercial calcium cyanamide (NEUBAUER) 7.

TOLLENS, B.: *Einfache Versuche für den Unterricht in der Chemie zur Unterweisung von studierenden Landwirten*. 4th Ed. revized and enlarged. Edited by P. Ehrenberg and B. Baule. Berlin: P. Parey. 100 pp. M. 16 + Teuerungszuschlag. For review see *Kolloid-Z.* 27, 271(1920).

Fertilizers. W. C. HOFFMANN. U. S. 1,360,401-2, Nov. 30. Fertilizers are prepd. by reaction of concd. or dil. H_3PO_4 on calcium cyanamide containing free lime.

Fertilizers. HEINRICH HEIMANN. Can. 206,568, Dec. 14, 1920. Fertilizers which can be easily distributed are made from fertilizing salts without drying by mixing a hot concd. soln. of NH_4NO_3 or melted NH_4NO_3 , which contains water with an inorg. salt.

Fertilizers. G. YOUNG and G. WATSON. Brit. 150,375, Apr. 29, 1919. A fertilizer in powdered form is obtained by burning or smoldering sewage sludge in a closed furnace under a gentle draft of air so regulated that, while H_2O , volatile oils and NH_3 are driven off, such combustion of the greases and other org. matter as takes place is complete and the formation of soot avoided. The sludge is partially dried before treatment. To prevent the temp. rising too high an excess of air is passed through the furnace. An inclined furnace that is rotary or through which the material is passed by agitators may be used. The NH_3 is recovered as sulfate and may be used to enrich the residue from the furnace. K_2SO_4 , superphosphates or humus may also be added to the residue.

Fertilizers. Soc. D'ETUDES CHIMIQUES POUR L'INDUSTRIE. Brit. 151,597, Sept. 2, 1920. A fertilizer containing variable amts. of N and phosphate is prepd. from cyanamide as starting material. The soln. of the latter is treated with an acid to convert it into urea or salts thereof, and then with an insol. phosphate, such as $\text{Co}_3(\text{PO}_4)_2$, bone ash, basic slag or natural phosphate, which is rendered sol. by the excess acid. By evapn. there is obtained a mixt. containing urea or salts thereof, sol. Ca phosphate and other Ca salts. According to an example, CaCN_2 in aq. suspension is treated with CO_2 , filtered, and the soln. treated with an excess of H_2SO_4 and heated to 70° . Bone ash or other insol. phosphate is then added and the whole evapd. to dryness. The CaCN_2 may also be decomposed by H_2SO_4 and the phosphate added without previous filtration.

Fertilizers. Soc. d'ÉTUDES CHIMIQUES POUR L'INDUSTRIE. Brit. 151,598, Sept. 2, 1920. Addition to 151,597 (preceding pat.). The process of prep. a mixed fertilizer containing N and phosphate described in the principal patent is modified by the use of H_3PO_4 or a sol. phosphate such as $\text{CaH}_2(\text{PO}_4)_2$ in place of H_2SO_4 for effecting the conversion of cyanamide into urea. The product is thus obtained free from CaSO_4 . According to an example, CaCN_2 in aq. suspension is decomposed by CO_2 , filtered, and the soln. treated with H_3PO_4 , followed by addition of insol. $\text{Ca}_3(\text{PO}_4)_2$, CaO , or CaCO_3 .

Fertilizers. J. R. PARTINGTON and L. H. PARKER. Brit. 151,024, June 26, 1918. NH_4NO_3 is mixed with bone meal. Other fertilizing ingredients which do not decompose the nitrate or the bone meal, such as K_2SO_4 or KCl , may be added. The NH_4NO_3 may be prep. by the process described in 136,190 (C. A. 14, 1417).

Potash phosphoric acid fertilizers. A. MESSERSCHMITT. Can. 206,574, Dec. 14, 1920. A mixt. of K-containing material, reactive phosphoric compds. and an amt' of reactive Cu compds. which contain at least 40% of CaO estd. upon the amt. of natural material are heated to a temp. slightly below the m. p.

Calcium cyanamide; fertilizers. AKT.-GES. FÜR STICKSTOFFDÜNGER. Brit. 150,979, Sept. 13, 1920. Granular CaNCN is produced by treating CaNCN with more than enough H_2O to slake the CaO , etc., under such conditions of temp. and pressure that the excess H_2O is immediately evapd. Temps. of $70\text{--}80^\circ$ and a reduced pressure are mentioned. The H_2O supply may contain dissolved or suspended matter which may enter into reaction with the CaO or may have a binding effect. The substances mentioned are Na_2SO_4 , FeSO_4 , clay, cyanamide and potato meal, and urea. CO_2 may be introduced with a salt soln. and may serve to spray the soln. over the cyanamide.

Fertilizers from organic material. A. J. GRINNELL and B. FIELD. Can. 206,399, Dec. 14, 1920. A substance is added to org. matter to neutralize its acidity, acid-producing and denitrifying bacteria are added and the whole is allowed to work by the growth of bacteria. A potash or phosphate compd. or both may be added after the addition of the bacteria.

Copper-containing composition for preventing damage to plants from parasites. G. MORSELLI. U. S. 1,362,172, Dec. 14. A compd. for treating plants, having the formula $\text{CaCl}_2 \cdot 3\text{CuO} \cdot x\text{H}_2\text{O}$, is prep. by slowly adding a 46°Bé. CuCl_2 soln., while cold, to milk of lime containing 15% Ca(OH)_2 , while constantly stirring, until only a slight excess of lime remains in the soln., filtering out the ppt. and washing and drying and disintegrating it to a fine powder.

Bacterial fertilizer. J. G. LIPMAN. U. S. 1,361,596, Dec. 7. A dry-fertilizing mixt. is prep. containing S 95–99.5% and 5–0.5% of a latent sulfofying bacteria culture. The latter may be prep. from S 100, phosphate rock 300, Fe sulfate 0.4, $\text{Al}_2(\text{SO}_4)_3$ 0.4 and fertile soil 1600 parts, by moist culture for 6–10 weeks followed by drying. CaSO_4 , CaCO_3 or peat may be used in similar mixts.

Phosphate and bacterial fertilizer. J. G. LIPMAN. U. S. 1,361,597, Dec. 7. A fertilizing mixt. is formed of 95–99.5% of dry pulverized phosphatic material such as phosphate rock and 5–0.5% of a dry culture of sulfofying bacteria, as described in the preceding pat.

Dicalcium phosphate. WALTER GLAESER. Can. 206,573, Dec. 14, 1920. Phosphate rock is heated with HCl and after the mixt. thus formed has cooled to 30° the liquid product is treated with Ca(OH)_2 to form amorphous CaHPO_4 sol. in NH_4 citrate. The ppt. is filtered and the remaining Ca phosphate-dried.

Calcium phosphate. W. P. THOMPSON. Brit. 151,684, June 14, 1919. Natural tricalcic phosphates are mechanically treated to obtain two fractions, one of rich granules containing a high % of phosphate, and the other of a mixt. containing CaCO_3 and

phosphate in so fine a state of division as to be assimilable by the roots of plants. The nodules of natural phosphate are first broken down and then treated in a rotary disintegrator through which a blast of air is directed. By this means, the granules of phosphate are freed from the carbonate dust which covers them and the mixt. is carried away to chambers where the two fractions sep. out according to d. Cf. 122,897 (C. A. 13, 1356).

Grinding and drying apparatus for fertilizers. A. J. CASE. Can. 206,422, Dec. 14, 1920.

Insecticides. W. H. ASTON. Brit. 150,758, Mar. 4, 1919. A liquid prepn. for attracting and destroying flies consists of a soln. of HCHO and an agent or agents attractive to the insects such as sugar, glucose, fruit sugar, molasses, fruit juices, or essences. A specified prepn. consists of formalin, glucose, and apple or pear juice.

17—PHARMACEUTICAL CHEMISTRY

W. O. EMERY

Advances in the chemistry of terpenes and essential oils during the years 1916-1919. A. RECLAIRE and F. ROCHUSSEN. *Chem.-Zig.* 44, 853-4, 865-7, 869-71, 875-6, 882-4, 894-6, 935-6(1920).—Numerous references accompany this review. E. J. C.

Essential oil of mustard—impurities and adulterations. WALTER DULIERE, Namur. *J. Pharm. Belg.* 2, 981-3(1920).—Artificial oil of mustard when prepd. from pure material (KSCN and allyl iodide) consists entirely of allyl isothiocyanate. Natural oil of mustard, obtained from black mustard seeds by steam distn. contains in addition to allyl isothiocyanate small quantities of C_2H_5CN and CS_2 . A sample of artificial oil of mustard examd. was found to be adulterated with 70% of EtOH and to contain about 5% of sulfides and polysulfides as impurities. The allyl isothiocyanate, isolated and weighed as thiosinamine, constituted only 16.5% of the total.

A. G. DuMEZ

Acriflavine. A. SCHAMMELHOUT. *J. Pharm. Belg.* 2, 997-9(1920).—This is a general article, in which the prepn., phys. and chem. properties, and uses of acriflavine are reviewed briefly. Acriflavine paste, prepd. by mixing 0.1% of acriflavine with Na stearate is the prepn. most commonly used in medical practice. Acriflavine gelatin and a starch mucilage containing 0.1% of the material are also used extensively.

A. G. DuMEZ

Matriculation sheets. A. SCHAMMELHOUT. *J. pharm. Belg.* 2, 857, 913, 929, 952, 970, 989-989, 1005(1920).—Tentative monographs proposed for introduction into the Belgian National Formulary are submitted for the following items: $CaBr_2$, CaF_2 , carmine, $Ca_3(PO_4)_2$, monobromated camphor, quinine and its salts as the hydrobromide, dihydrochloride, bisulfate and tannate.

A. G. DuMEZ

Ointments and solutions of liver of sulfur. A. LABAT. *Bull. soc. pharm. Bordeaux* 58, 226-32(1920).—An ointment for the treatment of scabies is prepd. by incorporating an aq. soln. of sulfuretted potash or soda with a base consisting of a mixt. of anhydrous wool fat and vaseline. Sulfuretted potash is preferable to sulfuretted soda as it is more readily sol. in the concn. used (1 in 2) and is more const. in compn. Sulfuretted potash as ordinarily prepd. consists almost entirely of K_2S_2 and $K_2S_2O_3$, whereas the soda prepn. contains, in addition to Na_2S_2 , 1 to 21.3% of $Na_2S_2O_3$ and traces to 21.21% of Na_2SO_4 . Only the freshly prepd. ointment should be dispensed, as the polysulfide in the portion exposed to the air is converted into thiosulfate on standing.

A. G. DuMEZ

Pharmaco-synthesis of organic antimony compounds. H. SCHMIDT. *Pharm. Ztg.* 65, 872-4(1920).—An address.

W. O. E.

Report on alkaloids. H. C. FULLER. *J. Assoc. Off. Agr. Chem.* 4, 156(1920).—Further work on the method for detg. atropine in tablets, is reported and a change in the former directions is recommended as follows: "Dry *in vacuo* to a const. wt. and weigh as atropine." The recommendations made were that: (1) The method for the detn. of atropine in tablets be changed as above indicated; (2) further work be done on the methods for sepg. quinine and strychnine, and a method be submitted to the collaborators, which has a reasonable certainty of yielding concordant results; (3) the members of the Assoc. submit to the Referee on Alkaloids suggestions for the study of important matters which may be developed in their practice. W. O. E.

Report on medicinal plants. A. VIKHOVVER. *J. Assoc. Off. Agr. Chem.* 4, 149-55 (1920); cf. *C. A.* 14, 3295.—The report is divided into 4 parts: (1) A method for the detn. of vol. oil in mustard seed and substitutes; (2) methods for the hydrolysis of linamarin and the subsequent detn. of HCN; (3) the effect of abnormal conditions on trade in crude drugs; (4) the value of wts. of unit vols. in the analysis of crude drugs and spices. It is recommended that: (1) The methods for the hydrolysis of linamarin and the subsequent detn. of HCN be adopted as tentative methods; (2) the method for the detn. of volatile oil in mustard seed and substitutes, especially with regard to the nature of the influence which alc. exerts if added before maceration, receive further study; (3) that new sources of supplies of proper substitutes for drugs not now obtainable be further investigated; (4) further work be done to det. the value of a more extended use of wts. of unit vols. in the analysis of crude drugs and spices. W. O. E.

Manufacture of thymol, menthone and menthol from eucalyptus oils. H. G. SMITH AND A. R. PENFOLD. *J. Proc. Roy. Soc. N. S. Wales* 54, 40-7(1920).—The individual constituents already detd. as existing in the oils derivable from the various species of *Eucalyptus* number about 40, among which are numerous alcohols, esters, aldehydes, terpenes, etc., but apparently only one ketone, namely piperitone (cf. H. G. Smith, *ibid* Oct. 1900, and *J. Chem. Soc.* 82, I, 108), quite generally distributed in certain *Eucalyptus* indigenous to the eastern and south-eastern portion of Australia and Tasmania, where the members of the whole group are generally known as "peppermints." The ketone is usually associated with the alcohol, piperitol. The broad-leaved peppermint, *E. dives*, appears to contain the ketone in largest amt., namely 40 to 50% of the oil, of which this species yields 3 to 4%. The remainder of the oil, largely phellandrene, is already recognized as being the best of all the essential oils for flotation work. For the manuf. of thymol, piperitone can easily be supplied in ton lots if required, and as the transformation to thymol can be effected in one operation, it would appear to represent a cheaper source for this phenol than even ajowan oil. Piperitone is an unsatd. ketone (*oxime* m. 110-1°, *oxamino-oxime* m. 169-70°, *semicarbazone* m. 219-20°, h. 229-30°, n_D^{20} 1.06-7°, d_4^{20} 0.9348, $[\alpha]_D^{20}$ -40.05°, n_D^{20} 1.4837, yielding on oxidation with $KMnO_4$ among other things isobutyric acid. On reduction with H in the presence of a Ni catalyst, piperitone yields menthone almost quantitatively, which on further reduction gives menthol. By heating with strong $AcOH$ in the presence of $FeCl_3$, thymol was obtained from the ketone in a yield of 25%. Methods can undoubtedly be devised whereby a theoretical yield would result. The menthol prepd. by reduction of piperitone in aq.- Et_2O soln. with Na followed by transition through the phthalic ester was optically inactive, thus following the general rule for synthetically prepd. substances. W. O. E.

Regulations for the control of arsphenamine, neoarsphenamine and sodium neoarsphenamine. M. W. IRELAND *et al.* U. S. Public Health Ser., *Misc. Publ.* 1920, No. 22.—These regulations, applicable to interstate traffic and to the District of Columbia, and designed as a guide for the manufacturer and control officials, embody very precise directions as to permissible containers, labels, Assent, and procedure to be followed in detg. the toxicity. W. O. E.

Solubility of vanillin and coumarin in glycerol solutions. M. DE GROOTE. *Am. Perfumer* 15, 372-4(1920).—The object of the investigation was to det. the solubilities of vanillin and coumarin in glycerol solns. of varying strengths over a range of 0.2 to 60.0°, such information being of considerable practical importance at the present time as related to flavors involving glycerol as solvent. The author's conclusions are embodied in the following summary: A method for detg. the soly. of vanillin and coumarin in glycerol solns. is described. Data are given showing the soly. of vanillin over a range of 0.2 to 60.0° for solns. containing up to 50% of glycerol. Similar data are given for coumarin. The immediate importance of this information and its application in actual manuf. are discussed. W. O. E.

Examination of artificial musk and other solid artificial perfumes. W. H. SIMMONS. *Perfumery Essentl. Oil Record* 11, 368-9(1920).—The various artificial musks, as also vanillin, coumarin and heliotropin, which had prior to the war reached a high standard, suffered during the war a very marked deterioration in quality, as indicated in the lowered melting points. From the results of comparative expts. given in 2 tables and involving 2 different melting pt. methods, it appears, first, that emphasis should be placed on the importance of detg. the m. p. as received, and second, that attention should be directed to the very inferior quality of much of the material of this class offered, not only during the war but at the present time. Quite satisfactory products can now be obtained if purity is insisted upon; but to claim, as is not infrequently done, that musk ambrette with a m. p. of about 60 to 90° is a "satisfactory sample of av. 'post war' quality" is only directly to encourage and perpetuate the production and sale of low-grade and inferior and even adulterated material. W. O. E.

Report on drugs. W. O. EMERY. *J. Assoc. Off. Agr. Chem.* 4, 148(1920); cf. *C. A.* 14, 3295.—Reference is had to work on the quant. analysis of monobromated camphor in migraine tablets, more specifically described in a previous paper (*C. A.* 13, 2104), as also to the examn. of arspenamine substitutes, the method studied in estg. the As being the one suggested by C. R. Smith (*C. A.* 6, 3381). W. O. E.

Chlorine in benzaldehyde. E. J. PARRY. *Perfumery Essentl. Oil Record* 11, 342(1920).—As the adulteration of natural oil of almond with BzH grew to its present huge dimensions, the presence of even the minutest trace of Cl (0.01% or less), being a definite index to the adulteration, became a matter of intense importance to those using it for this purpose. Since a BzH of such purity is Cl-free for all purposes other than the adulteration of natural oil of almonds, and particularly since any other product which is 99.99% pure is described as "pure," and traces of impurities which are of no importance are neglected, it is suggested that it would be entirely proper to describe such benzaldehydes as "Cl-free," when they contain these minute traces of Cl. This would cause inconvenience to those using it for adulteration, but to no one else. W. O. E.

Spanish oil of thyme. E. M. HOLMES. *Perfumery Essentl. Oil Record* 11, 338-9(1920).—Only a few authentic specimens of the no less than 30 species of *Thymus* enumerated in the flora of Spain have ever been distd. with the view to analyzing their volatile oils. Spanish oil of thyme has been imported for many years owing to its higher content of phenols than the French oils. Soon after the beginning of the war attention was directed to the fact that the Spanish product contained considerable thymol, further investigation showing that at least 2 species of Spanish thyme, *T. vulgaris* and *T. syris*, are capable of yielding thymol in paying quantity. It still remains to be demonstrated which yields the most thymol, and also which species of *Thymus* yields the oil containing carvacrol, problems well worth further scientific study. W. O. E.

Review of tests for methyl salicylate in oils of gaultheria and birch. CHARLES H. LA WALL. *Am. J. Pharm.* 92, 391-5(1920). W. G. GAESSLER

An improved method for the assay of aconite preparations. E. J. CHAPPEL AND NORA L. ALPFOY. *Am. J. Pharm.* 92, 822-3(1920).—See *C. A.* 14, 2964. W. G. G.

The theory of percolation. JAMES F. COUCH. *Am. J. Pharm.* 92, 770-96, 853-91 (1920).—All published knowledge on the subject of percolation has been classified, coordinated, and subjected to critical analysis. The results of every important contribution are presented and the best technic under varying conditions, as detd. by the experience of the whole body of pharmacists, is stated. Certain factors which bear on the problems of percolation and have not yet been investigated are discussed and attention is directed to other factors which require reinvestigation owing to the inadequateness of our present data. Explanations for a number of phenomena observed during percolation are offered and a discussion of the mechanism of extn. is included. A section of this survey deals with the various processes and forms of app. designed to solve some of the problems of simple percolation, especially such methods as repercolation and interrupted percolation. A comprehensive bibliography is appended. W. G. G.

Preferred base for zinc oxide ointment. TORALD SOLLMANN. *J. Am. Med. Assoc.* 75, 1420-2(1920).—From a questionnaire sent to the members of the Am. Dermatological Assoc., a majority of the replies were distinctly in favor of petrolatum as the basis of ZnO ointment. It was considered as equal to hard therapeutically, and superior in consistency, keeping quality and absence of irritation. It is therefore recommended that petrolatum be made the basis of the official ointment. L. W. R.

A method for alkaloidal assay. RAPP. *Pharm. Zentralhalle* 61, 551-5, 569-74 (1920).—An acid soln. of the alkaloid is prepd. in the usual manner. Fats and resins are removed by cooling to 0° and filtering, chlorophyll by repeatedly concg. an aq. soln. of the alkaloid on the H₂O-bath and filtering off the decompd. chlorophyll. The ext. having a vol. of 12-15 cc. is made alk. with NaOH or K₂CO₃, after the addition of 2 g. Na₂SO₄ and 15 g. gypsum are then added and the mixt. is shaken out with 25 cc. CHCl₃. More gypsum is added if the CHCl₃ does not sep. readily from the pasty mass. The 2d ext. is made with 20 cc. CHCl₃ after the addition of 5 g. gypsum and the 3d with 10 cc. CHCl₃ after the addition of 5 g. more of gypsum. The efficiency of the extn. depends on the total removal of H₂O by the gypsum. The CHCl₃ soln. is shaken out with 0.1 N HCl and titrated with 0.1 N alkali to a golden yellow, using Me red as an indicator. H. A. SHONLE

The evaluation of pepsin. M. BACHSTEZ. *Pharm. Zentralhalle* 61, 479-81(1920).—The method is based on the pptn. of protein from an acid soln. by the addition of AcONa. One g. pure casein is dissolved in 16 cc. of 25% HCl and the soln. made up to 1 l. To 10 cc. portions of this are added 10 cc. of a soln. containing the pepsin to be tested, starting with an initial amt. of 0.2 g. and decreasing to 0.00062 g. After digesting 15 min. 20 drops of a 20% soln. of AcONa are added. A ppt. forms in the tube containing undigested casein. This method can be used to test the activity of pepsin in pharmaceutical preps. H. A. SHONLE

The alkali and oil content of the seeds of the meadow saffron. CLEMENS GRIMM. *Pharm. Zentralhalle* 61, 521-4(1920).—Colchicine was present to the extent of 0.11-0.52%. The smaller seeds had the higher alkaloidal content. The actual amt. present was 1-25% greater than was indicated by physiol. activity. The seeds contain 6.6-8.4% of an oil having d_{20}^{25} 0.9176, congealing point -9°, n_{20}^{25} 1.4642, acid no. 20.32, sapon. no. 184.31, I no. 128.5 and contained 0.71% of unsapon. substance. The fatty acids have a congealing point of 22.5°, m. 24.0°, n_{20}^{25} 1.4646, neutralization no. 187.6 and I no. 131.0. H. A. SHONLE

The determination of hydrocyanic acid (MORRIS) 7.

ZÖRNIG, H.: Der Anbau von Arzneipflanzen. Edited by H. W. Frickhinger. München: Verlag von Natur und Kultur Dr. F. J. Völler. 112 pp. M. 6. For review see *Chem.-Zig.* 44, 855(1920).

Synthetic drugs. SOC. ANON. POUR L'INDUSTRIE CHIMIQUE À BAÏLE. Brit. 150,401, May 28, 1919. Allyl ester of 2-phenylquinoline-4-carboxylic acid is prepd. by allylating the acid, or a salt or halide thereof, by the usual methods. The product is tasteless and dissolves uric acid more readily than does the free acid.

Ointments. M. BRIEN, Nl. Brit. 151,134, July 25, 1919. An ointment is prepd. by dissolving Hg in HNO_3 , adding the soln. to a mixt. of solid petroleum and liquid petroleum, and finally adding small quantities of oil of cajuput and eosin. According to the Provisional Specification, vaseline is used in place of the solid and liquid petroleum.

Allyl *p*-aminobenzoate. R. ADAMS and E. H. VOLWILER. U. S. 1,360,994, Dec. 7, 1920. $p\text{-NH}_2\text{C}_6\text{H}_4\text{COOCH}_2\text{CH}=\text{CH}_2$ is prepd. as follows: A mixt. of mol. proportions of $p\text{-NO}_2\text{C}_6\text{H}_4\text{COCl}$ and allyl alc. is refluxed for 1-2 hrs. and then upon vacuum distn. yields allyl *p*-nitrobenzoate, a straw-colored oil *b*_p, 178°. The latter is reduced with Fe and HCl at a temp. of about 50°, the reaction mixt. is filtered from Fe, made alk. with NaOH and then extd. with ether. The ether soln. is evapd. and the product may then be crystd. from H_2O or petroleum ether. It m. 51-2°, and is a local anesthetic. Allyl 3,5-diaminobenzoate, a viscous liquid which does not solidify at room temp. is obtained by the reduction of allyl 3,5-dinitrobenzoate. It forms a dihydrochloride when dissolved in ether and treated with an excess of an alc. soln. of HCl. This dihydrochloride has a yellowish color and m. at 224° with decompn. The allyl 3,5-dinitrobenzoate used for prepg. this compd. is itself prepd. by heating an excess of allyl alc. with 3,5-dinitrobenzoic acid for an hr. under a reflux condenser, distg. off any excess of allyl alc. then remaining, and permitting the product to cool and solidify.

Acid sodium phenylcinchoninate. M. E. WOLVERAMP. U. S. 1,361,128, Dec. 7, 1920. Acid Na phenylcinchoninate of the formula $\text{C}_{10}\text{H}_{10}\text{NO}_2\text{NaC}_{20}\text{H}_{21}\text{NO}_8$, crystallizable in citron-yellow crystals freely sol. in H_2O , is made by trituration of phenylcinchoninic acid with an aq. soln. of a half mol. proportion of NaOH. The salt is sol. in H_2O and is a uric acid eliminant.

Vaccines. ELEKTRO-OSMOSE AKT.-GES. (GRAF SCHWERIN GES.). Brit. 150,328, Aug. 20, 1920. Specific vaccines for the active immunization of men or animals are prepd. by causing a continuous elec. current to act on emulsions of dead bacteria so that the cell contents are liberated by osmotic action. The living bacteria are killed by the action of an elec. current or otherwise and are then subjected to the action of a continuous current of medium strength and low voltage for several hrs. In an example, a current of 12 amp. at 15-25 volts is employed in app. having an effective electrode surface of 400 sq. cm. The process causes the cell contents to issue into the surrounding medium, and in most cases causes granular disintegration of the cell walls. For immunization, the clear ext. or the cell walls or residues, or a mixt. thereof, may be employed. The clear ext. may be concd. artificially. Specified vaccines are those obtained from the bacilli of swine fever, chicken cholera, and tuberculosis. Osmotic treatment of bacteria by simple immersion in H_2O or in very dil. or concd. salt solns. is also referred to.

Vaccines and immunizing serums. ELEKTRO-OSMOSE AKT.-GES. (GRAF SCHWERIN GES.). Brit. 150,334, Aug. 21, 1920. Addition to 150,328 (preceding pat.). Specific vaccines prepd. by the action of an elec. current on suspensions of bacteria, as described in the principal patent, are stabilized by the addition of a protective colloid. The added colloid may be derived from immune blood serum corresponding to the particular bacteria employed so that the resulting vaccines confer passive immunity in addition to the active immunity. The colloid added is preferably a completely sol. proteid, such as paraglobulin and albumin. In examples, normal blood serum or immune serum is mixed with bacilli sepd. from a culture medium or with the whole bouillon culture and placed in the center compartment of a three-cell electro-osmotic

app. divided by electrodes and diaphragms, and current is passed for several hrs., salt soln. being added as required to maintain the cond. After the required period the passage of current is continued to eliminate the electrolyte and to cause the sepn. of euglobulin. The clear soln. to be used as a vaccine is sepd. from the deposit of euglobulin and insol. bacteria residue in a centrifugal app. In some cases the sepd. deposit is dissolved in H_2O containing electrolytes and feebly alk., and is dild. to form a vaccine to be used alone or mixed with the original clear soln. The bacteria specified are those of swine fever and chicken cholera and streptococci, pneumococci, and meningococci.

Destroying microorganisms. ELEKTRO-OSMOSE AKT.-GES. (GRAF SCHWERIN GES.) Brit. 150,318, Aug. 19, 1920. Microorganisms are killed by the action of a continuous current of low voltage and comparatively large current strength. Liquids to be treated receive an addition of NaCl or other electrolyte and are placed in the center compartment of a three-cell app. comprizing a container divided longitudinally by diaphragms which carry on the sides remote from the center compartment electrodes of network or lattice work. In app. having an effective area of 400 sq. cm. a current of 10-12 amp. at 20-30 v. is employed. NaCl is added during the treatment to maintain the strength of the electrolyte which may be kept in circulation by stirring or by the introduction of inert gas. H_2O is circulated through the electrode compartments to regulate the temp. and to remove electrolytic products. At the conclusion of the process surplus electrolyte may be removed electroosmotically. Solids to be treated are satd. with an electrolyte and placed in the center compartment or are suspended in the electrolyte. Data are given as to the sterilization of pure cultures of various organisms and the sterilization of diphtheria serum without destroying its antitoxin content.

Vaccines. ELEKTRO-OSMOSE AKT.-GES. (GRAF SCHWERIN GES.) Brit. 150,318, Aug. 19, 1920. Addition to 150,318 (preceding pat.). The process described in the principal patent for killing bacteria by means of a current of electricity is applied to the treatment of pure cultures of various microorganisms for the prepn. of sp. vaccines. By treatment for a shorter period or with a smaller current the organisms are not killed but become non-pathogenic and may be used directly for the prepn. of vaccines or used directly for the prepn. of sub-cultures on artificial nutrients. These sub-cultures are also non-pathogenic and may be used for making vaccines containing the living bacteria. Bacteria to be treated are sepd. from their culture medium, emulsified in physiological salt soln. by continued shaking, and treated by the elec. current. Data are given for the treatment of swine fever bacilli, pneumococci, and tubercle bacilli. Known processes for the prepn. of vaccines by treating bacteria at a temp. of 58-60° or by means of disinfectants such as cresol and phenol are referred to.

18—ACIDS, ALKALIES, SALTS AND SUNDRIES

T. LYNTON BRIGGS

History of sulfuric acid production in Canada. J. BOWMAN AND A. NIEGHORN
Can. Chem. Met. 5, 14-6(1921). E. H.

The synthesis of ammonia at high pressures. GEORGES CLAUDE. *Chimie & Industrie* 4, 5-18(1920); *Bull. soc. chim.* 27, 705-24(1920); cf. C. A. 14, 1185, 1412.—
A review and discussion of the synthetic process of fixing N. W. H. ROSS

The history of helium and its production. R. T. ELWORTHY. *Can. Chem. Met.* 5, 7-9(1921). E. H.

Gypsum in 1919. RALPH W. STONE. U. S. Geol. Survey, *Mineral Resources of U. S., 1919*, Pt. II, 99-113 (Preprint No. 8, publ. Dec. 28, 1920). E. J. C.

ESCARD, JEAN: *Production industrielle synthétique des composés nitrés et leurs dérivés*. Paris: Dumod. 260 pp. 30 fr.

HACKER, W.: *Handbuch der Kitten und Klebemittel*. Meissen: Math. Bohlmann. 161 pp. M. 15.

Sulfuric and other acids. P. L. PFANNENSCHMIDT. Brit. 150,734, Sept. 6, 1920. Addition to 149,867 (C. A. 15, 405).—In the construction of acid chambers, towers, mains and other acid-proof structures as described in the parent pat. the sheets of glass, quartz glass, or the like are made with reinforced or thickened edges, which may be in the form of flanges, and the sheets are arranged so that the edges abut against one another while the supporting framework is outside, and need not therefore be of acid-proof material. A suitable construction is specified.

Nitric acid. C. L. TAYNTOR. U. S. 1,361,416, Dec. 7. A plant for manuf. of HNO_3 from NaNO_3 and H_2SO_4 is so constructed that all the stills of a 20-still acid house are provided with a common expansion chamber with 5 bleachers to which the expansion chamber delivers. Condensers from the bleachers deliver to a common chamber to receive uncondensed gas, adapted to deliver to a tower house. This arrangement equalizes pressure and facilitates uniform operation.

Nitric acid. N. C. HILL and H. L. MOYLER. U. S. 1,362,418, Dec. 14. HNO_3 is made continuously by maintaining a flow of hot H_2SO_4 gradually merging into a flow of molten sulfates, distributing alkali metal nitrate on the surface of the H_2SO_4 at a mid point in its flow and withdrawing HNO_3 vapors at a point nearer the origin of the acid flow. An app. is described.

Nitric acid. GENERAL ELECTRIC Co. Brit. 150,836, June 17, 1919. Oxides of N are absorbed in HNO_3 with a concn. from 60-70%, and cooled to a low temp., namely, in the region of 40° ; and free N and H collected as by-products at certain stages in the process are combined to give the liquid NH_3 used to cool the absorption chamber. A suitable construction is specified.

Nitric acid by the catalytic oxidation of ammonia. A. MASCHER. Ger. 307,052, Nov. 10, 1917. Instead of NH_3 gas, the vapors obtained by heating NH_4HSO_4 or $(\text{NH}_4)_2\text{SO}_4$ or mixts. of these salts, together with air, are conducted at an elevated temp. over catalyzers. The construction of a special generating app. for NH_3 and the employment of CaO are unnecessary. The entire H_2SO_4 content of the NH_4 salts is recovered as such, and the loss of NH_3 does not occur. The reaction $\text{NH}_3 + 2\text{O}_2 = \text{HNO}_3 + \text{H}_2\text{O}$ can be so carried out that H_2SO_4 containing H_2O and highly concd. HNO_3 can be obtained separately. The H_2SO_4 obtained by cooling in stages can be returned in part to the process.

Manufacture of nitric acid of highest concentration. M. MOEST and M. ECKARDT. Can. 206,819, Dec. 21, 1920. HNO_3 the percentage strength of which is higher than that of the acid of lowest vapor pressure is heated without additional substances, the vapors are passed through a fractionating column in which the temp. is kept at a degree corresponding with the desired concn. and the vapors passing over are condensed.

Concentrating dilute nitric acid. E. REINAU. Ger. 305,171, Sept. 30, 1916. After solid NaNO_3 is obtained from the dil. HNO_3 by admixt. with alkali sulfate (according to 299,001), and NaHSO_4 is obtained from the mother liquor by distn. (according to 299,007), both these products (NaNO_3 and NaHSO_4) are brought into reaction and the resulting Na_2SO_4 is returned to process for converting dil. HNO_3 to solid NaNO_3 and NaHSO_4 . The reaction proceeds as follows: $\text{HNO}_3 + x \text{H}_2\text{O} + \text{Na}_2\text{SO}_4 = \text{NaNO}_3 + \text{NaHSO}_4 + x \text{H}_2\text{O}$. $\text{NaNO}_3 + \text{NaHSO}_4 = \text{HNO}_3 + \text{Na}_2\text{SO}_4$.

Concentrating nitric acid. BAYER & Co. Ger. 310,081, Sept. 7, 1918. The required heat for the process is secured by employing strongly preheated H_2SO_4 and

utilizing the heat liberated by mixing dil. HNO_3 with the H_2SO_4 . The HNO_3 is driven off in a column app. which is charged with filling material and provided at the top with a vessel into which dil. HNO_3 and bot. concd. H_2SO_4 flow together. The temp. of the H_2SO_4 flowing in is maintained so high that the temp. of the acid leaving the column is at least 100° . Into the bottom of the column air is blown in amt. just sufficient completely to denitrate the acid flowing from the column. The H_2SO_4 employed is so concd., and the amt. of 40-50% HNO_3 added is such that the acid leaving the app. has a strength of 74%. In this manner, a HNO_3 of about 99.5% strength is obtained with a minimum amt. of air and without the use of steam.

Alumina; nitric acid. NORSKE AKTIESELSKAB FOR ELEKTROKEMISK INDUSTRI NORSK INDUSTRI-HYPOTEKBANK. Brit. 151,259, Aug. 27, 1920. A soln. of $\text{Al}(\text{NO}_3)_3$ is evapd. to distil off dil. HNO_3 , and H_2O , steam, or fresh soln. is added so as to keep the temp. constant (about 140°) until the bulk of the Al_2O_3 is pptd. as a cryst. basic nitrate which is practically free from Fe even if the soln. is ferruginous. It is advantageous to treat solns. containing $\text{Ca}(\text{NO}_3)_2$ or NaNO_3 , which remains in soln. in the mother liquor. Such solns. may be obtained by adding some base or salt such as limestone to the nitrate soln. before heating. Alternatively the soln. of nitrate to which such a base or salt has been added, or from which some acid has been distilled off may be heated under pressure in an autoclave to effect the pptn. of the basic nitrate. The basic nitrate obtained as above described is heated to obtain Al_2O_3 and N oxides which are converted into HNO_3 . The mother liquor is treated with HNO_3 to convert the Al_2O_3 content to normal nitrate and the soln. is evapd. until $\text{Al}(\text{NO}_3)_3$ crystallizes out and may be returned to the original soln. and the remaining mother liquor is evapd. to dryness to recover the NaNO_3 or $\text{Ca}(\text{NO}_3)_2$.

Recovering acids or metallic salts. H. E. TIFFANY. Can. 206,749, Dec. 21, 1920. The materials used to harden fiber are removed by placing the satd. fiber with the hardening material in an electrolyte and passing a current through it.

Alkali sulfates; hydrochloric acid. T. GOLDSCHMIDT, AKT.-GES. Brit. 150,962, Sept. 9, 1920. In the production of alkali sulfates and HCl by Hargreaves' process, the briquets of NaCl are fed continuously down a shaft which receives at its lower end a continuous stream of SO_2 and O, any temp. adjustment that may be necessary being effected outside the shaft. A suitable construction is specified.

Ammonia. SOCIÉTÉ L'AIR LIQUIDE (SOC. ANON. POUR L'ÉTUDE ET L'EXPLOITATION DES PROCÉDÉS G. CLAUDE). Brit. 150,744, Sept. 7, 1920. To prevent undue rise of temp. in the catalytic synthesis of NH_3 under high-pressure (particularly hyperpressure) conditions, only a portion of the gases to be treated is heated to the reaction temp. before passage into the catalytic material, the remainder being introduced therein in the cold condition and in regulable quantities. The mixt. of N and H enters the catalytic chamber by a pipe; a portion passes through the heat interchanger into the catalytic material where it intermingles with the remainder of the mixt. introduced directly into the material through the valved pipes; the product escapes by another pipe. A suitable app. is specified.

Cyanamide. WARGONS AKTIEBOLAG and J. H. LIDHOLM. Brit. 151,583, June 30, 1920. Cyanamide practically free from dicyanodiamide is obtained in soln. by supplying successive quantities of CaCN_2 to H_2O or a cyanamide soln. while supplying enough CO_2 to maintain the soln. neutral or only slightly alk. By starting with a cyanamide soln. very strong solns. can be obtained. The reaction is effected at a temp. not above 70° , and the CO_2 is preferably supplied under pressure, being blown into the soln. by nozzles; or other means of bringing the gas into intimate contact with the liquid may be employed, such as heaters; or the soln. containing CaCN_2 may be sprayed into an atm. of CO_2 . The CO_2 may be pure or mixed with other gases, as, for instance combustion or lime kiln gases.

Cyanides. C. T. THORSELL and H. L. R. LUNDEN. Brit. 151,098, June 20, 1919. In the production of cyanides by the reaction of free N or a mixt. of an alkali or alk.-earth metal or compd., C and a catalyst, the catalyst such as FeMn, Ni or Cu is introduced in the form of a compd. not containing any element other than C, H, O and N, and is reduced at a temp. below 550°. Fe(OH)₃ prepd. by pptg. FeSO₄ with NH₃, and Fe oxalate are mentioned. Cf. 3,341, 1878, 1,004, 1880, 5,617, 1881, and 116,365.

Cyanides; retorts. E. C. R. MARKS. Brit. 150,127, June 23, 1919. In the production of cyanides by a process such as that described in 13,332, 1914 (C. A. 9, 3121), in which briquets are fed down a vertical retort, clogging of the charge is prevented by making an abrupt increase in the diam. of the retort at a point below the heating zone where the charge becomes tacky. The retort may be made of telescopic sections, the heating section being preferably of a refractory alloy such as an Fe-Ni or an Fe-Cr-Ni-Mn alloy and provided with a flange to support its wt. The retort is removable from the furnace, the brickwork being removable, and is bolted to a receiving and conveying app., the joint being packed with kieselguhr or the like.

Mixing or comminuting and dissolving alkali silicates. F. J. PHILLIPS and E. J. ROSS. Brit. 151,508, Nov. 28, 1919. In mixing or comminuting and dissolving alkali silicates by agitating the partially disintegrated silicate in the presence of H₂O under the action of steam or gas in a rotary mixer, the steam or gas is arranged to be directed in the form of jets throughout the whole internal space of the mixer and to impinge with force upon the particles of the material treated. A suitable construction is specified.

Neutral ammonium sulfate. P. PARRISH and W. A. M. VALON. U. S. 1,360,765, Nov. 30. Neutral ammonium sulfate is produced by condensing the vapors from a fixed ammonia still, condensing vapor withdrawn under reduced pressure from the waste liquor from the fixed ammonia still, mixing the 2 condensates so as to obtain a liquor of the correct content of NH₃ and spraying sulfate crystals to be neutralized with the liquor.

Magnesium carbonate. B. B. GRUNWALD. U. S. 1,361,324, Dec. 7. Calcined magnesite is hydrated with H₂O and the liquid mass thus obtained is subjected to the action of CO₂ under pressure to produce MgCO₃ and Mg(HCO₃)₂ in the form of a milky mixt. Calcined magnesite is added to the mixt. as it comes from the digester with an excess of CO₂ in soln. and the mixt. is agitated and heated to ppt. basic Mg carbonate.

Apparatus for producing magnesium carbonate from calcined magnesite. B. B. GRUNWALD. U. S. 1,361,325, Dec. 7. The app. comprises a hydrating chamber to which a supply of CO₂ is connected, a heated pptg. device and other devices adapted for carrying out the method described in U. S. 1,361,324 (preceding pat.).

Purifying titanium hydrates. G. JENSEN. U. S. 1,361,866, Dec. 14. Undesirable acid substances are removed from pptd. Ti hydrates by treating the pptd. material with BaCl₂ or other similarly acting metal compd. which forms neutral and relatively insol. salts with the acid substances. U. S. 1,361,867 relates to a similar process in which the pptd. Ti hydrates are freed from acid impurities by the addition of NH₄OH or other similarly acting alk. substance which forms neutral sol. salts which are removed.

Ammonium chloride. SALZWERKE HEILBRONN, AKT.-GES. Ger. 308,354, Oct. 5, 1916. NH₃ or gases containing NH₃, in the presence of steam, is allowed to act on alkali or alk. earth chlorides heated above 150°, or fused. CO₂ may also be present. A portion of the HCl may be expelled first by means of steam alone, before the introduction of NH₃. The addition of Fe powder, Fe₂O₃, Al₂O₃ and the like expedites the reaction. The action of these substances is due apparently to the formation of intermediate products, and is catalytic.

Molybdenum compounds. G. RICHTER. U. S. 1,360,581, Nov. 30. Mo compds. such as Na molybdate are produced from wulfenite by treating the material with H_2O and a sulfurizing agent such as Na_2S and then treating the soln. thus obtained with additional wulfenite or other desulfurizing agent. The wulfenite residue from the last step is used for continuing the process with Na_2S soln.

Carbonyl chloride. S. PEACOCK. U. S. 1,360,312, Nov. 30. Carbonyl chloride is made by passing a mixt. of air and Cl over carbonaceous material such as coke at a temp. of about 1200-1500°. The $COCl_2$ may be sepd. from N by condensation.

Table salt. C. DIETZ. U. S. 1,362,087, Dec. 14. A table salt is prepd. by coating granules of rock salt with NaCl deposited on the granules by treatment with a pure satd. NaCl soln.

Apparatus for manufacture of salt. R. MURAMATSU and C. ÔTA. Japan 35,894, March 1, 1920. The bottom of the preheating furnace is inclined so that muddy substances will sep. from the salt soln.

Mercuric oxide. C. BRUSA and V. BORELLI & Co. Brit. 150,917, Oct. 30, 1919. HgO is obtained by decomposing $HgNO_3$ by heat in a closed chamber; the N oxides evolved are brought into contact with Hg in presence of O to yield more $HgNO_3$ for use in the process. A suitable construction is specified.

Tin oxide. D. DE ROS. U. S. 1,362,237, Dec. 14. A body of Sn is heated to an oxidizing temp. (preferably about 1000°) and the heating is then discontinued and hot air or other oxidizing gas is supplied and exothermic oxidation is effected to produce SnO_2 which is volatilized and condensed.

Hydrogen. S. W. BRAY and I. H. BALFOUR. U. S. 1,360,876, Nov. 30. In producing H by the action of H_2O vapor on Fe, the whole of the gases which have effected reduction in one of the units of the app. are passed from that unit, after they have become non-reducing, through a scrubber and condenser for removing moisture, CO_2 and S compds. and the purified gas is then passed into another unit containing an unreduced charge to effect reduction of the latter.

Decolorizing carbon. R. VON OSTREJKO. U. S. 1,362,064, Dec. 14. In the manuf. of decolorizing carbon, the retorts containing the charcoal are externally heated to redness by a furnace and superheated steam is then admitted to the charcoal in the retorts to liberate combustible gases by the action of the steam on the charcoal. The combustible gases are led from the lower portion of the retorts to the furnace for combustion in the latter to supply heat thereafter to the retorts. A portion of the partially prepd. charcoal is discharged from the retorts into the furnace and the calcination of the charcoal is finally completed in the furnace with exclusion of air.

Enriching dilute nitrous gases obtained by air combustion. NORSK HYDRO-ELEKTRISK KVAELSTOFARTSELSKAB. Ger. 318,091, Sept. 24, 1916. The dil. nitrous gases are absorbed in H_2SO_4 of about 90%. The absorbed N oxide gases are then expelled by heating this mixt. with the employment of a dephlegmating app. which is fed at the top with the mixt. The N oxides are combined as nitrosylsulfuric acid in accordance with the equation $2H_2SO_4 + N_2O_2 = 2HNO_3 + H_2O$. The claim is made that mixts. of $NOHSO_3$ and H_2SO_4 can be practically completely freed from N oxides by simply heating them, if an acid not stronger than 90-91% be used for the absorption.

Treating monazite sand. E. L. KNOEDLER. U. S. 1,361,735, Dec. 7. A mixt. of monazite sand and H_2SO_4 is heated and then poured directly from the vessel in which it has been heated into a drowning tank to facilitate handling.

Aluminium compounds for sizing paper and like purposes. G. MURK. Ger. 319,420, May 14, 1919. Unburned minerals containing Al, such as clay, bauxite and the like, are treated with H_2SO_4 or solns. of alkali bisulfates in the presence of H_2F_2 , its salts or other F compds. E. g., 100 parts of powdered unburnt clay or other Al

mineral, and 5 parts powdered fluorspar are introduced, successively, into 200 parts cold H_2SO_4 of about 40° B ϕ . or into the corresponding amt. of NaHSO_4 soln. of about the same strength, contained in an open vessel provided with a stirring mechanism. The contents of the vessel are then kept in motion for a long time. The mass may be heated finally in order completely to decompose the clay. The H_2F_2 liberated by the H_2SO_4 readily attacks the clay to form F-Al compds. which are immediately decomposed by the excess H_2SO_4 to form H_2F_2 or SiF_4 , which then break down additional clay.

Alumina; abrasives. CARRORUNDUM Co. Brit. 150,116, June 18, 1919. A fused Al_2O_3 for abrasive purposes, containing from 0.2–1.3% of TiO_2 and less than 0.8% each of Fe_2O_3 and SiO_2 , is obtained by fusing in an elec. furnace a mixt. of aluminous material such as bauxite, clay, emery, etc., with enough C to obtain a product containing 90% of Al_2O_3 , cooling, crushing, sepg. metallic particles, and again fusing the product with a small quantity of C sufficient to obtain the further reduction of impurities necessary to obtain the production of the above-stated compn. Instead of using for the second fusion the product obtained by the first described treatment, waste or dust from Al_2O_3 which has been fused for abrasive purposes may be used, but in all cases C is employed in the final fusion. The product is used for abrasive purposes such as grinding wheels, and is preferably agglomerated by ceramic material. A suitable compn. is abrasive grains 80%, Albany clay or other fusible clay 14%, stone-ware clay 5%, and feldspar 1%. This mixt. is burnt at 1350°.

Plastic compositions. J. F. LEGER. Brit. 2,341, Jan. 29, 1914. Walls, linings, panels, roof coverings, vehicle bodies, etc., are formed by applying metal trellis or wirework to the frame of the structure and coating the trellis with a plastic non-combustible material which is also a non-conductor of heat and sound. The material consists of the following proportions by wt.: MgO 1, Canadian asbestos fibers $1/2$, corkwood $1 1/2$, wood pulp 1, common asbestos $1/2$, talc, $1/4$, and BaSO_4 , all mixed with sufficient MgCl_2 of 15–30° B ϕ . to bind them. The plastic material is applied with a trowel or brush to both sides of the trellis and may afterwards be planed, polished, and varnished.

Plastic compositions. F. RINGER. Brit. 151,641, Sept. 27, 1920. Animal or vegetable fibers are soaked in a soln. of MgCl_2 , a small quantity of a fatty acid or glycerol is added, coloring matter as desired, and finely powdered soapstone is mixed in, and finally powdered magnesite is added with stirring until a plastic mass capable of being poured is obtained. The mass is molded under high pressure to form buttons, boxes, handles, vessels, wall coverings, and the like. In an example, the proportions of the various ingredients are as follows: 10 g. cellulose fibers, 350 cc. 20% soln. of MgCl_2 , 20 g. of glycerol, 60 g. soapstone, 700–800 g. MgO powder.

Cleaning and polishing composition. H. T. NICHOLS and C. R. FREEDR. U. S. 1,362,260, Dec. 14. A compn. for cleaning and polishing painted and varnished surfaces is formed of gasoline and a heavier mineral oil such as "Coilar fuel 14+."

Metal polishes. A. L. WARBRICK. Brit. 151,078, June 13, 1919. A polish for Ag, brass, and Cu articles consists of 1 part KCN, $1/4$ part AgNO_3 , 2 parts whiting, 1 part H_2O . Equiv. quantities of other sol. Ag salts and cyanides may be used.

Cleaning wire. BRITISH THOMSON-HOUSTON Co. Brit. 151,399, July 10, 1919. Carbonaceous, oxide, and other impurities formed upon wire during its manuf., particularly wire used for *elec. lamp filaments and supports*, is removed by treating the wire in a chem. soln., to loosen the impurities and then subjecting the wire to a scrubbing treatment. The soln. used may be of NaOH or K_2CO_3 , or preferably of borax heated to about 98°. A suitable app. is specified.

Hardened molded casein product. H. P. BASSETT. U. S. 1,380,356, Nov. 30. Casein is pptd. in the presence of a hardening agent such as CH_2O ; the ppt. is dried until it contains 18–20% moisture and is then molded under heat and pressure. The

product thus formed does not shrink, warp or discolor. Fillers, coloring matter, etc. may be incorporated, preferably during the pptn.

Adhesives. O. V. FOURZ. U. S. 1,380,915, Nov. 30. A paste adhesive suitable for general use is formed of glue 2 oz., tapioca 2 oz., NaCl 0.5 oz., glucose 0.5 oz., and H₂O or vinegar 14 oz.

Rendering tacky surfaces temporarily non-adhesive. J. A. NOBLE. U. S. 1,361,894, Dec. 14. Tacky surfaces such as cloth coated with a rubber or tar mixt. are temporarily rendered non-adhesive by the application to the surface of a solid adhesive material such as pulverized glue or modified starch. This treatment facilitates handling of the material, e. g., in the manuf. of a camera bellows.

Fireproofing aircraft. P. R. BRADLEY. Brit. 151,398, July 8, 1919. Cloth and wooden parts of aircraft are fireproofed by coating them with alginic acid or alginates, preferably after applying cellulose acetate or nitrate dope. A suitable construction is specified. Cf. C. A. 14, 212.

"Artificial slate." E. FAHRIG. U. S. 1,362,563, Dec. 14. A mixt. adapted for making roofing tiles or tool handles is formed of powdered slate 41, SiO₂ 11, S 15, "magnesite" (MgO?) 10, talc 9, kaolin 8, and sericite 6 parts, mixed with AlCl₃ or MgCl₂ soln.

19—GLASS, CLAY PRODUCTS, REFRACTORIES AND ENAMELED METALS

G. E. BARTON, C. H. KERR

The ceramic industries. EDWARD W. WASHBURN. Univ. Ill. *Tech. Eng. News* 1, No. 8, 1(1920).—A brief discussion of the field covered by the ceramic industries, their importance in modern life, the need of research, and the opportunities which exist for the technically trained man in this industrial field.

C. W. PARMELEE

Electric furnace refractories. A. F. GREAVES-WALKER. *Chem. Met. Eng.* 23, 933-38(1920).—A brief description is given of raw materials now available for highest grade brick, together with a list showing recommended refractories for melting the different metals and alloys, and practical hints on the care, storage and laying of roofs, hearths and linings. Only the cryst. form of magnesite is suitable for refractories. That of Washington is inferior to the Austrian variety, because of high SiO₂ and low Fe content. The use of chromite bricks has been almost discontinued, due to poor domestic ore. The limitations of zircon, C, dolomite, zirkite, carborundum, alundum, bauxite, fireclay and SiO₂ refractories are discussed. Selection of refractories depends upon (1) product of furnace; (2) whether basic or acid process; (3) type of furnace; (4) intermittent or continuous operation. Refractories recommended for electric furnaces: (1) *Steel Ingots and Castings*: Magnesite for high grade steels; SiO₂ where no special requirements are demanded. (2) *Alloy Steels*: Magnesite exclusively. (3) *Ferromanganese*: Magnesite or carbon. (4) *Ferrochrome*: Magnesite or chrome. (5) *Ferrosilicon*: SiO₂. (6) *Cast Iron*: Fireclay for low temp.; SiO₂ for high temp.; magnesite for low sand requirement. (7) *Copper*: Magnesite on bronze scrap. SiO₂ or magnesite on refined pig. (8) *Bearing Metal*: Magnesite low in Silica. (9) *Monel Metal*: Magnesite preferably, silica can be used. (10) *Bronze*: SiO₂ preferably, magnesite may be used. (11) *Lead*: Magnesite exclusively. (12) *Silver*: SiO₂. (13) *Al and Alloys*: Magnesite exclusively. Special care must be taken in construction of the roof. Spalling is excessive between skewback and electrode rings. This can be partly overcome by use of bricks of special shape (the Griffin patent). These bricks have an offset near the upper side which prevents grout from penetrating, leaving the expansion joint free to give. At least 1/16 inch per foot should be allowed for expansion of magnesite bricks.

O. A. HOSSEN

Porcelain insulation for high-voltage transmission systems (STILL) 4.

LOGAN, W. N.: Kaolin of Indiana. Indianapolis: The Dept. of Conservation, State of Indiana, Division of Geology. 132 pp. For review see *Chem. Met. Eng.* 23, 1138(1920).

Tonindustrie-Kalender 1921 (in 2 parts). Berlin: Verlag der Tonind.-Ztg. G. m. b. H. M. 8.50 for both parts. For review see *Tonind.-Ztg.* 44, 1287(1920).

Compositions and processes for coating glass surfaces. GENERAL ELECTRIC CO. Brit. 150, 598, Jan. 8, 1920. The glare of elec. light may be overcome by applying to the globe a coating consisting of Na silicate, French chalk, H_2O , and a pigment, and then, when dry, covering with a waterproofing mixt. of dammar varnish made very thin with turpentine. To give the appearance of opal glass, ZnO is used as the pigment and the paint is made up with rather less H_2O than when colored pigments, such as sienna, are used. The glass surface may be sand-blasted before the first coating is applied, and in place of French chalk other forms of hydrous Mg silicate may be used. Designs can be produced by dissolving portions of the first coating with acids, or by other means, and then covering with the varnish.

Refractory material. O. HUTCHINS. U. S. 1,362,316, Dec. 14. A refractory material suitable for furnace linings, crucibles or muffles is formed of a burnt mixt. of zirconia and alumina.

Refractory material. O. HUTCHINS. U. S. 1,362,317, Dec. 14. A mixt. of burnt zirconia and chromite is used as a refractory material for furnace construction and similar uses.

Refractory composition. F. J. TONG. U. S. 1,362,274, Dec. 14. A highly refractory compn. adapted for glass or metal working furnaces is formed of a burnt mixt. of zirconia and sillimanite.

Basic refractory composition. H. P. BASSETT. U. S. 1,360,355, Nov. 30. A basic refractory compn. suitable for lining open-hearth furnaces or Bessemer converters is formed of magnesian limestone 100, Fe scale or oxide 1-2, NaCl 2 and SiO_2 2-10 parts, heated to a high temp. and prepd. in granular form.

Enamels. G. RUPPRECHT. U. S. 1,360,585, Nov. 30. In making enamels containing Zn orthosilicate from raw materials such as ZnO and SiO_2 , the materials are melted by direct action of a high-temp. flame while protected from contamination by extraneous substances and supported during the melting on an unmelted portion of the raw materials.

White enamel on steel. S. RIESER. U. S. 1,360,317, Nov. 30. Frit for use in producing a white enamel on steel is ground with H_2O and clay and a portion only of the sol. enameling constituents is removed. This serves to form a base which is used with Na antimonate, Sn oxide and Sb sulfide. U. S. 1,360,318 relates to the production of an unmottled white enameled steel vessel or surface with a single application of a coating material directly on the surface of the metal. The coating may be formed according to the method of the preceding pat.

20—CEMENT AND OTHER BUILDING MATERIALS.

C. N. WILBY

Fused cements and the electric furnace. J. BIED. *Rev. l'ingénieur* 27, 191-3' (1920). Fused aluminous cements have approx. the formula: $SiO_2 \cdot 2CaO + 2(Al_2O_3 \cdot CaO)$. These cements react to a very slight extent with sulfate waters. After 72 hrs. the strength of the mortar is equal to that of any artificial cement after three months. The curve of hardening of cement is not a parabola as is commonly assumed,

but is a branch of the hyperbola. By plotting time against the inverse of the strength, or time against the settlement of the tested beam an equilateral hyperbola of the equation $(l - a)t = C^2$ is obtained, where l represents the inverse of the strength or deflection of the beam and a is a coeff. different for each cement. By use of the above formula it can be shown that the strength of hydraulic cement does not increase indefinitely with time, but is limited by the ordinate of the asymptote. Tests have shown that aluminous cement made in the elec. furnace is cheaper and more uniform than that made in a water-jacketed furnace fed with coke. B. believes that fused aluminous cement marks an important date in the history of reinforced concrete.

J. A. MONTGOMERY

Marble resources of Alaska (BURCHARD) 8.

FRTSCH, J.: *Fabrication du ciment*. 2nd Ed. revized. Paris: Librairie scientifique et médicale, 93, Boulevard St.-Germain. 550 pp. 45 fr. For review see *Mon. sci.* 10, 216(1920).

Reinforced concrete. A. T. J. GUERITTE and L. G. MOUCHEL & PARTNERS Brit. 150, 880, July 28, 1919. Concrete structures exposed to sea water are reinforced by bars which present a mechanical bond with the concrete and have a protective coating to resist corrosion. The coating may be varnish or other compn. applied by dipping the bars or by painting or spraying, or the bars may be oxidized, bronzed, or otherwise chemically treated to preserve them.

Molding polished slabs, etc. A. WEITHALER. Brit. 150,326, Aug. 20, 1920. Relates to the production of slabs or other articles with polished surfaces from cement-stone compns., and consists in molding the compn., smoothing the surface, exposing the molded article to damp air for some time, and pressing by means of a bright or polished tool. The time of exposure may be reduced by introducing small quantities of the salts of fatty or other saponifiable acids into the compn.

Coating compositions. C. H. IVINSON. Brit. 151,666, Apr. 2, 1919. An anti-corrosive compn. which may be used to impregnate a cement or concrete layer to form a damp-proof course, consists of asphalt dissolved in a volatile solvent such as benzene or gasoline, oleates or stearates of Ca, Mg, Al, or other metals, and gum elemi. Masticated rubber, gutta-percha, paraffin wax or colophony and filling materials such as asbestos, silica, powdered glass, graphite, Fe_2O_3 , ZnO, or other oxide may be added.

Road-surfacing material. G. L. COLLORD. U. S. 1,361,140, Dec. 7. Road-surfacing material is formed in lumps consisting of adhering hygroscopic and non-hygroscopic materials such as produced by mere superficial reaction of HCl on crushed slag, limestone or dolomite.

Slaking lime in a vertical container. C. E. CANDLAT. Ger. 323,519, Sept. 26, 1913. The H_2O used for slaking the lime is introduced within the CaO mass itself and in an amt. which corresponds to the lime discharged from the container.

Lime and like kilns. W. SOMERVILLE. Brit. 150,157, July 23, 1919. A lime or like kiln is provided with air inlets above the fire and with one or more uptakes communicating with the interior of the kiln below the burning zone, the draft being downwards, while the CO_2 or other gases pass away through the uptakes, draft being induced by air which enters at the foot of the kiln and is heated by the hot lime before entering the uptakes. Induced or forced draft may be used to assist in carrying off the CO_2 . A suitable construction is specified.

Treating wood. I. DE VECCHIS. Brit. 151,661, Mar. 3, 1919. Wood is treated with a soln. of double sulfates or chlorides of Cu, Zn, and Mg with NH_4 used simultaneously, preferably in conjunction with free alkali, to enable it to resist parasites and

to increase its strength. The wood is placed in a closed container in which a vacuum is produced, and may be subjected to heat to facilitate the removal of the sap. The treating soln. is then allowed to be sucked into the container, and is subsequently pumped in until a pressure of 8-14 atm. is obtained. After the necessary time has elapsed the wood may be removed or may again be subjected to a vacuum in a hot or cold state prior to allowing it to dry in the open air. Cf. 5,893, 1900, and 9,989, 1900.

21—FUELS, GAS, TAR AND COKE

J. O. PENNOCK

Analyzing of coal by the Bureau of Mines. F. G. COTTRELL. *Elec. Rev.* 78, 75(1921).—Brief outline of the work of the bureau and suggestions for better and more economical utilization of coal. C. G. F.

A multiple bomb calorimeter for coal testing. S. B. BILBROUGH. *J. S. African Assoc. Anal. Chem.* 3, 4-5(1920).—Six calorimeters are observed simultaneously. The readings for 6 can all be made in the time occupied by a detn. so that time is saved and the work is more interesting. W. P. WHITE

By-product coke, anthracite and Pittsburgh coal as fuel for heating houses. HENRY KREISINGER. *J. Ind. Eng. Chem.* 13, 31-3(1921).—Comparative tests of three fuels, anthracite with a calorific value of 12,636 B. t. u., coke with 11,756 and a Pittsburgh coal with 13,239, were made with house boilers fired at 50%, 80% and 125% of their ratings. Results under the three conditions were: for the coke, av. heat absorbed 7975, efficiency 68.1%; anthracite, absorbed 8440, efficiency 66.8%; coal, absorbed 7313, efficiency 52.2%. It should be noted that the anthracite used was superior to that now on the market, having an ash content of only 11.56%, hence the results given are rather high. Data on house-heating tests with coke and Pittsburgh coal are also given. H. L. OLIN

The technical importance of lignite and bituminous coal. FRITZ HOFFMANN. Breslau. *Brennstoff Chem.* 1, 25-6(1920).—Abstract of a review of recent fuel research. W. B. V.

Combustion of naphthalene solutions in internal-combustion engines. L. S. PALMER. *Iron and Coal Trades Rev.* 101, 789(1920).—Abstract. Some results are given of expts. carried out to obtain an efficient, cheap fuel of high calorific value, and to investigate the possibility of the efficient combustion of solid fuels by means of soln. P. discusses the heat values of different solns. of naphthalene and benzene and describes the tests undertaken with a motorcycle to det. the consumption of these solns. The results show that the mileage per gal. increased from 104.9 mi. for pure benzene, to 107.8 mi. for a 15 to 25% soln. of naphthalene with benzene. A 15% soln. was the highest that was found practicable without choking the carburetor with a solid deposit of naphthalene. It is concluded that (1) naphthalene dissolved in benzene to a strength of 15% by wt. forms an efficient fuel for ordinary gasoline motors, and though about 3 d. per gal. cheaper yields 3.4% more mileage than com. benzene; (2) a soln. rather than a mechanical mixt. forms an efficient method by which solid or heavy liquid fuels may be burned conveniently in ordinary internal-combustion engines. Such a soln. had a calorific value about 3.3% higher than benzene alone. J. L. WILEY

Manufacture of motor spirit in gas works. J. NICHOLLS. *Gas World* 73, 403-4 (1920).—Abstract. Gas works in England ought to recover 100 million gals. of benzene annually. The operation of recovery of benzene was described in detail in the original article. Washing the gas with tar has not been wholly satisfactory as the tar was too

viscous at high temp. in winter for scrubbing towers; the amt. of tar produced from 1 ton of coal was only 11 gals. with which to wash 12,000 cu. ft. of gas; and the max. efficiency was only 50% as compared with 80-90% by oil washing. Blast-furnace oil had been used as a diluent but had the disadvantage of having paraffin present. Paraffin oil had been used satisfactorily and anthracene oil was extensively used. Since paraffin had already been used as a motor fuel, there appeared to be no objection to a little paraffin appearing in motor fuel, hence washing of vertical-retort gas might be profitably undertaken. The cost of production of motor fuel was put at 12 d. per gal.

J. L. WILBY

Benzene as a substitute for kerosene in lighting. WENDT. *J. Gasbel.* 63, 653-4 (1920); illus.—Four types of lamps using benzene as the lighting medium are described.

J. L. WILBY

Determination of the calorific value of the volatile constituents of coal as a measure of its suitability for gas manufacture. R. MEZGER AND M. MÜLLER. *J. Gasbel.* 63, 669-73 (1920).—A simple method is described, with a diagram, for detg. the heating value of the volatile constituents of coal and typical analyses are shown. It is an improvement on the method of Strache and Hiller (*Ibid* 54, 994; 59, 129). The method consists primarily in heating 0.2-0.5 g. of coal in a quartz tube sealed at one end, the other end being connected with a Hempel gas buret. The tube is contained in an insulated furnace heated to and maintained at 1000°. The tar is sepd. and collected on asbestos wool filling a space in the tube which is cooled by water. The tar-free gas is cooled to atm. temp., measured and analyzed by known methods and its calorific value calcd. by known methods or in a Strache calorimeter (*C. A.* 5, 1212). It was found that the calorific value of the volatile products was less than that calcd. by subtracting the calorific value of the coke from that of the original coal. It is concluded that loss of heat took place due to exothermic reactions during gasification.

J. L. WILBY

Comparative heat efficiency of generator-gas firing and pulverized-coal firing. E. TERRES. *J. Gasbel.* 63, 673-5 (1920).—A lignite coal containing moisture 53.63, C 27.04, H 1.72, O 11, N 0.39, S 0.79 and ash 5.43%, and having a calorific value of 2500 cal. per kg. was dried to 15% moisture content and pulverized. When fired in this condition with an excess of air from 1:1 to 1:3, a limiting temp. of 2050° to 1850° resp. was obtained. The gas used for the 2nd test was obtained by carbonization and complete gasification of lignite. Carbonization of 100 kg. of lignite produced 20 cu. m. of gas containing CO₂ 22, heavy hydrocarbons 1, CO 14, H 48, CH₄ 10 and N 5%, and 119 cu. m. of producer gas containing CO₂ 7, CO 25, H 10 and N 58%. The mixed gas analyzed CO₂ 9.17, heavy hydrocarbons 0.14, CO 25.43, H 15.46, CH₄ 1.4 and N 50.4% and had a heating value of 1230-1320 cal. per cu. m. On firing, this gas with the same amt. of excess air as above gave a temp. of 1625-1480°. By pre-heating the air to 850°, this figure was increased to 1960-1810°. The comparative thermal efficiencies of gas firing and pulverized-lignite firing are 53.29% and 75.73%, the difference being the heat required to convert the lignite into gas.

J. L. WILBY

Facts regarding gas standards. J. B. KLUMPP. *Gas Age* 46, 506-12 (1920).—Abstract of the report of the Am. Gas Assoc. Comm. on calorific standards. From an economic standpoint, the best gas for the future should be a mixed coal and water gas produced with a minimum of oil and through complete gasification of the coke obtained from the coal gas manuf. Its calorific value should not exceed 500 B. t. u.

J. L. WILBY

Modern developments in the manufacture and utilization of coal gas. F. J. WISER AND T. A. TOMLINSON. *Gas World* 73, 444-6 (1920); *Gas J.* 132, 576-7 (1920).—Some particulars are given of the carbonization of coal in continuous vertical retorts and the industrial use of gas.

J. L. WILBY

Apparatus for saving gas with gas burners. K. BUNTE. *J. Gasbel.* 63, 714-6 (1920).—Some simple appliances for saving gas with domestic gas burners are described.

J. L. WILBY

Massachusetts low B. t. u. gas tests. L. J. WILLIEN. *Gas Age* 46, 515-7(1920).—From the Am. Gas Assoc. report on gas standards. In 1918 the gas standard was changed from 16 c. p. to 528 B. t. u. It was found from census of several companies that this drop from the av. of 600 B. t. u. did not affect appreciably the consumption of gas. It did, however, reduce the consumption of gas oil about 15%. J. L. W.

The addition of carbonic acid gas to the generator process. O. ESSICH. *Feuerungstechnik* 8, 184-5(1920).—Attempts at solving the problem of improving the generator process in past years resulted mostly in failures (for records of these attempts see Fischer's Year Book 1890, p. 190 and 1893, p. 233).—The increasing price of fuel demands the solution of the problem. There is no doubt that there is a certain amt. of advantage in the addition of furnace gases containing CO_2 to the vaporization atm. E. discusses advantages and disadvantages of the process and offers suggestions for suitable expts. The following advantages are anticipated: (1) Greater evolution of gas per kg. of fuel; (2) reduction of steam demand; (3) consequent reduction of steam production; (4) better adaptation of the gases for metallurgical purposes (Martin furnace); (5) production of warm waste gases by the addition of hotter waste gases. At generator temp. of 500-1000° there is an addition of 1 cc. CO_2 gas to 5 cc. O, both entering at same temp. The waste gases are hot and theoretically the CO_2 content of the mixt. can be increased up to 0.5% for each 100° temp. of waste gases. A disadvantage is the danger that through lowering of temp. in the generator chem. equil. will be shifted and a higher CO_2 content will appear in the gas. Only a moderate CO_2 increase, however, is anticipated. It is pointed out that the CO_2 addition must be const. If the permissible amt. be exceeded the generator cools too much and poor gas results. The use of a regulator is recommended. The distribution over the cross section of the pit must always be correct. The addition of waste gases is not permissible in the reduction zone but must be made in the upper half of the combustion chamber.

H. C. PARISH

Calorific value and the efficiency of gas burners. J. H. DAWE. *Illuminating Engineer (London)* 13, 252(1920).—Some of the results of using low-grade gas are discussed. A series of photographs showing the effect on mantles supplied resp. with gas of 290, 468 and 635 B. t. u. indicated that the illumination with incandescent mantles was the same; with flat flames, the flame was much smaller and less luminous with the low-grade gas. The new condition involving use of low-grade gas will favor the use of incandescent burners to the exclusion of flat-flame.

N. VAN PATTEN

Classification of the technically combustible gases. H. R. TRENKLER. *Feuerungstechnik* 8, 157-9(1920).—Six classes are recognized: (1) Natural gas; (2) rich gas; (3) poor gas; (4) full gas; (5) oil gas, and (6) inert gas. Subspecies are as follows: Class 1 is subdivided into marsh gas and fire damp. Under 2 come distn. gas and coke gas. Types of coke gas are coke-oven gas and illuminating gas. Subdivisions of 3 are air gas such as that from Siemens, Martin and blast furnaces, semi-gas, Mond gas and regenerator gas, the last being typified by blast-furnace regenerator gas and that from lime-kiln regenerators. Water gas belongs to class 4. Examples of 5 are Aerogene gas, Blau gas, Pentans gas and Benoid gas. Carbogas is also given as a variety of oil gas, Carbo-water gas and Oltier gas being mentioned as examples. The general methods of prepn. of each variety are discussed briefly.

H. C. PARISH

The behavior of mine gases at high temperatures, alone and in admixture with the other constituents of a fire-damp explosion. H. WINTER. Bochum. *Brennstoff Chem.* 1, 17-22(1920).—Mixts. of methane (purified mine gas, of approx. 80% CH_4 ,

20% N_2) and air or O_2 in various proportions were exploded in a Hempel pipet and the resulting gases analyzed. Results may be summarized as follows:

$CH_4 + O_2$		$CH_4 + \text{air}$	
Ratio O_2 to CH_4	% CH_4 burned to CO_2 and H_2O	Ratio O_2 to CH_4	% CH_4 burned to CO_2 and H_2O
1.876	71	2.028	95.5
1.756	61	1.881	88
1.661	50	1.637	60
1.422	34	1.526	51
1.189	16		
1.029	12		

The other constituents of the products of combustion were CO , H_2 and often small quantities of C and CH_4 , the latter apparently arising from the equil. of CO_2 and H_2 rather than incomplete combustion. In CH_4 -air mixts. of 5.0-9.2% CH_4 , the combustion is essentially complete; from 9.2-14% CH_4 , the products of combustion always contain CO . The max. of the explosion lies at 9.2% CH_4 rather than 9.5%, since a ratio of O_2 to CH_4 of 2.06 is required for complete combustion, rather than the theoretical 2.0. The paper includes a full bibliography and discussion of the earlier work on the combustion of CH_4 and on the various gaseous equilibria involved. W. B. V.

Enrichment of artificial gas with natural gas. JAMES B. GARNER. *J. Ind. Eng. Chem.* 13, 58(1921).—A discussion of the project of enriching manufactured gas with natural gas and the applicability of the various manufactured gases for the purpose. Blue water gas is recommended as the best manuf. gas for the purpose. Less than 80 cu. ft. of natural gas has an enriching value of one gallon of gas oil. F. W. P.

Experiences with a Tully plant at Keltly. H. RULE. *Gas World* 73, 488-90 (1920); *Gas J.* 152, 704-6(1920).—The Tully water gas plant is used in conjunction with a horizontal retort plant. Its advantages over other types are: (1) low first cost as compared with verticals or horizontals; (2) ease of meeting sudden demand; (3) requires less manual labor than horizontals; (4) S compds. are less; (5) capacity per shift per man double that of horizontals; (6) low upkeep; (7) reduction in costs for labor and coal. The plant and its operation are fully described. The process is simple in working, requiring only ordinary intelligent regulation of steam during the progress of the run and as the temp. in the generator diminishes to keep the CO_2 content of the gas at a fairly low and const. level. The gas made per ton of coal is 46744 cu. ft. of 330 B. t. u. of a very uniform quality. An analysis shows CO_2 4.6-5.4%, O_2 0.2-0.3, CO 36.1-38.1, unsatd. hydrocarbons 0.4-0.6, H_2 and CH_4 49.2-50.6, N_2 7.0-7.5, total inerts 12.3-12.7. The coal gas in the mixt. is 537 B. t. u. and the mixed gas consists approx. of $\frac{1}{2}$ coal gas and $\frac{1}{2}$ Tully gas representing 12,250 cu. ft. of a 470 B. t. u. gas analyzing CO_2 4.3%, O_2 1.8, CO 16.5, unsatd. hydrocarbons 2.4, CH_4 and H_2 63.4, N_2 11.6, total inerts 17.7. The large % of O is due to its admittance in excess in order to keep the purifiers clean. The operation results are entirely satisfactory with great economy. J. L. WILEY

Water gas generator with steam producer. W. NIEHUS. *J. Gasbel.* 63, 713-4 (1920).—For small and medium-sized gas works, which do not have a stationary boiler in operation, a water gas generator with automatic steam producer is especially suitable. It does not require special attention and the fuel consumption is appreciably less since steam is generated by use of waste heat. Such a plant is described. J. L. WILEY

Air purge system in water gas sets. G. H. SMITH. *Gas Age* 46, 502-5(1920).—S. reports after practical experience that production efficiency is improved through adoption of the air purge in operating water gas sets. It consists in blasting the set for 5 to 15 secs. after the steam purge at the end of a run thus freeing the set of blue gas, any remaining oil gas and any of the volatile matter from the coal left in the set

and injecting this lean gas into the relief holder instead of wasting it by burning at the stacks. The advantages to be gained are (1) a saving of generator fuel, (2) an increased capacity of the set; (3) a saving of generator steam; (4) an easier handling of temp. control, and (5) no increase in amt. of oil used per 1000 cu. ft. Operating results show for a 7' 6" and a 8' 6" set resp. an increase in make per sq. ft. of grate area per hr. of 41 and 19.5%; a saving of generator fuel per 1000 cu. ft. of 29 and 16.5%; a saving of generator steam per 1000 cu. ft. of 25% in either case; a slight increase in B. t. u.; and an increase of 2 and 5% in consumption of gas oil per 1000 cu. ft. This latter was due to a lowered efficiency of the gas oil from 81.7 B. t. u. per gal. to 79.1 B. t. u. rather than to the use of the purge.

J. L. WILEY

Determination of the partial and total pressures of aqueous solutions of $\text{NH}_3\text{-CO}_2$ and their application to ammonia scrubbing and the concentration of ammoniacal liquor. E. TERRES AND H. WEISER. *J. Gasbel.* 63, 705-12(1920).—Complete details of method and app. are given for detg. the partial pressures of water vapor, NH_3 and CO_2 in proportions corresponding to various solns. of $(\text{NH}_4)_2\text{CO}_3$ at temps. of 20, 40, 60.8 and 80°, and the respective total pressures at these temps. and at 90 and 95°. The former were detd. by an aspiration method, the latter by a manometric method giving values closely corresponding to the sums of the partial pressures. It was not possible with the app. to det. the partial pressures for solns. higher than 8.46% $(\text{NH}_4)_2\text{CO}_3$ at temps. above 80°. The results are as follows:

PARTIAL PRESSURE IN mm. Hg.

$\text{NH}_3\text{-CO}_2$ soln. calcd. as %		H ₂ O.				NH ₃ .				CO ₂ .			
$(\text{NH}_4)_2\text{CO}_3$		20°.	40°.	60.8°.	80°.	20°.	40°.	60.8°.	80°.	20°.	40°.	60.8°.	80°.
2.82	16.8	55.6	155.0	350.0		1.8	4.8	15.3	35.8	2.7	9.6	53.5	106.0
5.65	16.3	55.0	153.2	348.0		3.6	9.7	27.0	75.0	3.7	17.6	82.2	156.6
8.46	15.6	54.2	153.5	345.0		5.1	14.5	38.9	112.3	4.4	22.0	100.5	200.5
11.28	15.4	53.1	152.2		7.1	19.4	51.0	4.9	24.0	118.1
14.10	14.8	52.0	151.0		8.8	24.4	62.8	5.2	25.3	129.5
16.92	14.2	51.0	150.0		10.6	29.5	72.5	5.4	26.6	137.2
19.74	13.8	50.2	149.0		12.3	34.6	84.0	5.2	27.4	152.0
22.56	13.3	49.3	148.0		14.2	39.5	102.0	5.5	28.0	163.1

TOTAL PRESSURE IN mm. Hg.

$\text{NH}_3\text{-CO}_2$ soln. calcd. as %		20°.	40°.	60°.	80°.	90°.	95°.
$(\text{NH}_4)_2\text{CO}_3$		2.82	21.0	68.0	215.0	500.0	801.0
		5.65	24.0	84.0	246.0	575.0	978.0
		8.46	25.0	89.0	278.0	680.0	1076.0
		14.10	28.5	100.5	331.0	854.0
		22.56	32.5	116.0	401.0

J. L. WILEY

Centrifugal ammonia washer at the Kiel-Wilk gas works. BARENFANGER. *J. Gasbel.* 63, 693-4(1920), illus.—A vertical centrifugal gas washer of 150,000 cu. m. capacity with an efficiency of removal of NH_3 down to a max. of 2 g. per 100 cu. m. of gas has been installed. It operates on the countercurrent principle and is driven by gear drive from an elec. motor at 350 r. p. m. The water consumption is 6 cu. m. per day. In an 11-day test with a daily throughput ranging from 49,000 to 63,400 cu. m. of gas with a NH_3 content between 0.36 and 0.69 g. per 100 cu. m., the wash water showed a NH_3 content of 2.66 to 4.13%. In another test a throughput of 57,700 cu. m. of gas yielded to the wash water 2.27% NH_3 leaving only 0.47 g. in 100 cu. m. of gas after the washer. The gas from the washer is tar-free, the small, re-

maining traces of tar after the separators being removed in the first chamber. The gas and water enter at a temp. of 13° and leave at 18°, and 15-16° resp. Such a washer could be used also as a benzene washer by circulating wash oil. J. L. W.

Neutral sulfate of ammonia by the Wilton process. ANON. *Gas J.* 152, 636 (1920); cf. Evans, C. A. 15, 303.—The process applies very well to plants making 2-6 tons of sulfate per day. The cost of a complete installation is about £300. NH_3 liquor with about 10-15% NH_3 is made by mixing mother liquor with milk of lime and heating with steam, the liberated NH_3 being absorbed in a closed tank. The pure NH_3 is run into cold mother liquor making it slightly alkaline. From 2-4 gals. of this liquor will neutralize 3-4 cwt. of sulfate in the centrifugal. The smell of pyridine is evident in this process but it may be reduced to a minimum by feeding acid continuously to the saturator. It is most evident in cases where the tar has not been entirely sepd. from the liquor in the wells. There can be no H_2S nor HCN as the source of the NH_3 makes it impossible. J. L. WILEY

The economy of gas producers for obtaining low temperatures and making ammonium sulfate. E. ROSE. *Stahl u. Eisen* 40, 387-95(1920).—The paper discusses the cost of production and is of interest to a chemical engineer. W. T. H.

Experiences with the direct recovery of sulfate of ammonia. A. G. CHRAL. *Gas J.* 152, 515(1920); *Gas World* 73, 452(1920); *Chem. Age (London)* 3, 624-5(1920).—C.'s experience with the direct process has been generally satisfactory. At one plant the av. production of sulfate over 5 yrs. was 21.6 lbs. per ton of coal. He found that satisfactory working was due to the conditions under which the plants work rather than to the type of plant. Efficient condensation of the gas before entering the acid washer has an important bearing on the yield. With inefficient condensation, the tar fog brought down in the acid washer prevents active absorption of the NH_3 and also gives trouble in its removal from the mother liquor before evapn. There is also the necessity for continuous circulation of the mother liquor so as to prevent stratification in the acid washer. A very probable source of NH_3 loss occurs in working up the sulfate soln. from the original 6-10% of H_2SO_4 down to 0.5% before evapn. To prevent this a large excess of washing area must be provided. Purifier trouble often occurs when the temp. of the NH_3 vapors delivered to the acid washer from the still is too high. This can be obviated by installing an efficient water-cooled condenser. Efficient operation demands considerable supervision throughout the process. The quality of the salt made by this process is favorable; an av. test gives 22.16% NH_3 , 2.2% moisture and no free acid. J. L. WILEY

What is the source of the benzene in coke-oven and gas retorts? II. FRANZ FISCHER AND HANS SCHRADER. Mülheim. *Brennstoff Chem.* 1, 22-4(1920); cf. C. A. 15, 162.—From the standpoint of the recently discovered influence of the presence of H_2 on the thermal decompn. of aromatics, namely, causing dealkylation and dehydration in preference to more deep-seated changes, F. and S. criticise the results of earlier workers, e. g., Schultz, on the decompn. of turpentine (*Ber.* 10, 113(1877)) and Krämer (*Ann.* 189, 129(1877)) and Müller (*J. prakt. Chem.* [2] 58, 1(1896)) on the decompn. of phenols. The various reactions of the type mentioned, namely dealkylation of benzene homologs, dehydration and reduction of phenols and splitting of polynuclear aromatics, are represented as being equilibria, the extent to which they proceed being detd. by the proportion of H_2 , CH_4 and H_2O in the gaseous products. The stability of the C_6H_6 homologs in the presence of H_2 decreases rapidly as the mol. wt. increases. Aniline is 90% decomposed to C_6H_6 and NH_3 when led with H_2 through the authors' tinned iron tube at 770°. The behavior of diphenylmethane and fluorine, biphenyl and carbazole, stilbene and phenanthrene, supports Haber's rule that the —C—C— structure in aromatic compds. is more stable than —C—H, while in the aliphatics the reverse is true. W. B. V.

Low-temperature carbonization and its application to high-oxygen coals. S. W. PARR AND T. E. LAYNG. *J. Ind. Eng. Chem.* 13, 14-7(1921).—The Parr low-temp. coking process for high-O coals involves the following fundamental principles: (1) Maintenance of temps. within the true low temp. range, *i. e.*, below the point at which the primary decompn. products, which are in the main condensible tars and oils, undergo a secondary decompn. with the formation of gases chiefly. This critical temp. is near 750°. (2) Regulation of the process to prevent the decompn. products of the oxygenated constituents of the coal from reacting with the "phenol sol." substance, which is unsatd., of definite m. p. and on decompn. furnishes the binder for the coke. Failure to accomplish this results in oxidation of the phenol-sol. and destruction of its cementing properties. (3) Control of temp. in such a way as to enable the exothermic reaction which occurs principally in the range of 300-400° to supply heat to the interior of the retort charge without the application of temps. in excess of those previously mentioned. Failure in this respect would result in "green" cores due to the insulating properties of coke. In marked contrast to the product of common coking practice, coke made in this way from Illinois coal of high O content or even from badly weathered samples is dense and firm. Yields of tar oil range from 20 to 30 gals. per ton, gas about 80% of that from high temp. processes, and combined N nearly equal, although not all is in the form of NH_3 . H. L. OLIN

Carbonization of Canadian lignite. EDGAR STANSFIELD. *J. Ind. Eng. Chem.* 13, 17-23(1921).—Work done by the Canadian Dept. of Mines shows that a Saskatchewan lignite with H_2O content of 35% and a calorific value of 7200 B. t. u. can be transformed to a fuel of 75% greater value by means of drying and carbonizing processes. At a max. temp. of 555° product percentages were (dry basis): water of decompn. 11.7%, gas 17%, tar 4.1% and carbonized residue 66.7%. Based on moist coal, gas yield per ton was 3130 cu. ft. (385 B. t. u.), $(\text{NH}_4)_2\text{SO}_4$ 10.2 lbs., tar 5.3 gals. and residue 910 lbs. The carbonizing app. is described in detail. H. L. OLIN

Commercial realization of the low-temperature carbonization of coal. HARRY A. CURTIS. *J. Ind. Eng. Chem.* 13, 23-6(1921).—See C. A. 14, 3519. H. L. OLIN

Carbonization in 1920. THOS. SETTLER. *Gas J.* 152, 602-4(1920).—Results are given of studies made on c. p. and calorific value of gas, control of working temps. in vertical retorts, and penetration of heat through the charge. The usual speed of penetration ranges between $\frac{1}{16}$ in. and $\frac{1}{2}$ in. per hr., and with down-steaming $\frac{1}{4}$ in. may be obtained. J. L. WILEY

Low-temperature carbonization. F. FOERSTER. *J. Gasbel.* 63, 621-8(1920).—F. traces the development of the low-temp. process in Germany, shows its importance from an economic standpoint and reviews some of the exptl. work carried out in the Institut für Kohlenforschung in Mülheim, with special reference to the work done by Fischer, Glund, Schneider *et al.* J. L. WILEY

By-product coking. F. W. SPERR, JR. AND E. H. BIRD. *J. Ind. Eng. Chem.* 13, 26-31(1921).—A general discussion of the subject of by-product coking with special reference to newer tendencies in utilization of by-products. H. L. OLIN

Modern coke plant at Birmingham. J. M. HASTINGS, JR. *Gas Age* 46, 461-5 (1920); cf. C. A. 14, 2852.—The Semet-Solvay coke oven plant of 120 ovens at the Sloss-Sheffield Steel plant is now furnishing city gas to the city of Birmingham, Ala. For the 5 months that the plant has operated the results av. as follows: Tons of coal charged per day 1764, volatile matter in coal 29.09%, yield dry coke (furnace) 72.52%, breeze 2.62%, sulfate (as liquor) 28 lbs., tar 9.1 gal., light oil 3.13 gal. J. L. WILEY

Effect of burners and bars on hot-plate efficiency (LARKIN) 1. The Sugg-Larkin hot plate (ANON.) 1. Centrifugal extractors and separators applied to the chemical industry (BROADBENT) 13. A historical contribution relative to the combustion

process (v. LIPPMANN) 2. Natural gas resources available in central north Texas (SHAW, PORTS) 8. Ignition of gases at reduced pressures by transient arcs (THORNTON) 24.

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Fuel. J. ARMSTRONG. *Brit.* 149,449, May 20, 1919. Briquets of fuel are formed with grooves, penetrating their side surfaces, to receive material adapted to expand under the action of heat and burst the briquet into pieces during combustion. The briquets are molded from a mixt. of materials, such as coal, peat, and sawdust with tar or other binder. A suitable construction is specified.

Liquid fuel mixture. J. BLACK. U. S. 1,360,872, Nov. 30. A homogeneous and permanent liquid fuel mixt. adapted for use in automobile engines is formed of 100 gals. of distillate produced by distg. a mixt. of an aliphatic hydrocarbon liquid (such as kerosene) testing not to exceed 50° Bé. with approx. 10% C_4H_{10} (the distillate having a higher Bé. test than the liquid from which it was obtained), mixed with gasoline 5 gals., ether 16 oz., acetone 100 oz. and alc. 14 oz.

Liquid fuel for motors. A. HAYES. U. S. 1,361,153, Dec. 7. A homogeneous fuel mixt. adapted for use in internal-combustion engines is formed of a petroleum distillate such as gasoline or kerosene 15, C_4H_{10} 7-20, MeOH or EtOH 9-70 and Me_2O or Et_2O 1 part.

Treating carbonaceous materials. W. E. TRENT. *Brit.* 151,236, Apr. 15, 1920. Pulverized coal thoroughly mixed with H_2O is treated with liquid hydrocarbon, e. g., crude oil, etc., to agglomerate the carbonaceous particles into a heavy greasy mass and exclude ash-forming residues and H_2O . Various quantities of H_2O and oil may be used. A suitable construction is specified.

Treatment of peat. WM. B. BOTTOMLEY. *Can.* 206,416, Dec. 14, 1920. Peat is moistened with a soln. of NaCl to develop nucleic acid derivs. therein; It is then heated under conditions to retain the water and inoculated with microorganisms. The temp. is maintained at a point suitable for bacterial growth.

Drying peat, etc. T. ROBERG and TECHNO-CHEMICAL LABORATORIES, LTD. *Brit.* 150,807, June 6, 1919. Peat and other substances are dried in two stages; first, on the surface of a drum heated internally by the vapor arising from the substance, the temp. of which has been raised by compression, as described in 150,068 (*C. A.* 13, 564), with or without the addition of steam from other sources; second, in a current of hot air, furnace gases, or the like. A suitable construction is specified.

Peat briquets; pumping plant. E. P. T. R. ULMANN. Brit. 149,889, Nov. 6, 1919. In the manuf. of peat briquets, the peat mass is disintegrated and mixed with H_2O to form a homogeneous peat porridge in a multistage screw pump, which also forces the mixt. through a jointed pipe line directly to a drying area over which it is spread in a thin layer and afterwards cut up by suitable means. A suitable construction is specified.

Cracking heavy oils from distillation of bituminous coal. C. S. PALMER. U. S. 1,360,973, Nov. 30. In order to increase the yield of light or volatile compds. obtainable from heavy oils resulting from the distn. of bituminous coal, the material is digested under a pressure of about 4 atm. (the pressure being mainly autogenous) and at a temp. above 200° for a period (usually a few min.) sufficiently long to induce cracking without causing sufficient carbonization seriously to interfere with the process. No aq. vapors are added during the digestion. The process is especially adapted for treating tar oils or pitches in order to obtain hydrocarbons of low b. p.

Cracking hydrocarbons derived from gas tar. M. MELAMIN. U. S. 1,362,127, Dec. 14. Gas tar containing relatively heavy hydrocarbons is distd. with steam and H_3PO_4 in order to convert heavy hydrocarbons into more volatile products adapted for use as a motor fuel. The treatment may be repeated to increase the yield of low-boiling products.

Purifying coal gas. D. MARBAIS. Brit. 151,463, Sept. 13, 1919. In a continuous process for removing naphthalene from coal gas by washing with anthracene oil at a temp. of 20° , and circulating the oil through a distg. app., as described in 118,734, both the gas and the oil are maintained constantly at temps. of about $18-22^\circ$ resp. The gas passes through a cooler to reduce its temp. to 18° and enters a packed tower supplied with anthracene oil at a temp. of about 22° ; the oil is satd. with benzol to prevent absorption of benzol from the gas. The oil leaves the washer by a pipe and passes to the distg. app., and the purified hot oil is passed through a cooler and returned to the washer. The H_2O from the gas cooler is circulated through the oil cooler.

Purifying gas. H. L. DOHERTY. U. S. 1,360,734, Nov. 30. Acid gases are removed from the high-pressure natural gas and other gas under high pressure without disturbing the pressure by scrubbing the gas under pressure with a flowing body of a H_2O and NH_3 , removing the scrubbing liquor, heating the liquor to expel absorbed acid gases, removing NH_3 from the acid gases and returning it to the liquor, cooling the liquor and returning it and the contained NH_3 into scrubbing contact with the gas in a closed-cycle system.

Purifying gases from the dry distillation of coal and recovering by-products. K. BURKHESER. Can. 206,812, Dec. 21, 1920. The gas is treated with S and a soln. of NH_4OH (from the gas itself) or other alk. reagent to remove H_2S ; the alk. soln. is regenerated with Fe oxide. The S sepd. from the gas is converted into SO_2 or SO_3 and then employed to combine with the NH_3 in the gas.

Producer gas. HARRY A. GRINE. Can. 206,377, Dec. 7, 1920. Liquid fuel, an O-carrying medium and steam are introduced into a gasifying chamber in such proportions that when the mixt. is ignited in the chamber, the steam cooperates with the fuel and O medium to form two zones, one of them a volatilization zone wherein the temp. is below the ignition point of the fuel but above its volatilization point and the other a combustion zone wherein the temp. rises above the ignition point and partial combustion takes place to form gas.

Producer gas. W. B. CHAPMAN and D. MASON. U. S. 1,361,137, Dec. 7. Fuel from which the producer gas is to be made is led into a heated gasifying chamber and the resulting gas is withdrawn from the chamber and subjected to the action of an ionizing medium such as elec. corona discharges while the gas is still hot from the

gasifying operation. This serves to facilitate sepn. of soot from the gas for return to the gas-producing zone.

Producing gas. LINDON W. BATES. Can. 205,182, Dec. 7, 1920. A stable mobile fuel comprizing liquid hydrocarbon and pulverized solid carbonaceous material is atomized into an ignited mixt. of air and steam in a generator heated to a temp. sufficient to transform the solid particles and the liquid hydrocarbon into gas, the ash is removed and the gas washed to remove tar and lampblack.

Producing gas. CRESTES U. BEAN. Can. 206,183, Dec. 2, 1920. Hydrocarbon and water are separately heated to about 780° F., the O is removed from the steam by the action of Fe, and the hydrocarbon and H are passed through a conduit at rising-temp. conditions until gasification takes place at about 1208° F., when the C and H combine into a fixed gas.

*Gas producer. T. CLOUSTON. U. S. 1,362,559, Dec. 14.

Gas producer for mechanical traction. JNO. WELLS. Can. 206,507, Dec. 14, 1920. Water tuyères are placed in or near the fuel space of a gas producer so that water vapor used in the producer is generated by the heating of the tuyères.

Producer for generating gas from coal. J. H. CORTHESY. U. S. 1,380,503, Nov. 30. Returned gas is introduced tangentially into the producer so as to whirl the fuel which is in pulverulent form.

Gas producer conduits. PHILIP D'H. DRESSLER. Can. 206,523, Dec. 14, 1920.

Coking. A. G. A. CHARPY. Brit. 150,996, Aug. 14, 1920. A coke oven comprises two or more chambers maintained at different predetd. temps. through which the charge is passed. The charge may be compressed during its passage through the oven and the gases evolved from the sep. chambers may be mixed in any proportions.

Regenerative coke oven. L. WILPUTTE. U. S. 1,360,609-10, Nov. 30. Structural features.

22—PETROLEUM, ASPHALT AND WOOD PRODUCTS

F. M. ROGERS

A new method for the determination of sulfur in oils. C. E. WATERS. *J. Ind. Eng. Chem.* 12, 482-5(1920).—Supplementary note. *Ibid* 612.—A preliminary report on Bureau of Standards *Tech. Paper* 177 (cf. following abstract). F. W. PADGETT

Sulfur in petroleum oils. C. E. WATERS. *Bur. Standards, Tech. Paper* 177, (1920).—The origin of S in petroleum, forms in which S occurs, identification and significance of S compds., detection of S and its compds., and the detn. of S in petroleum. Under the last mentioned heading is described a new method (cf., however, Goetzl, *Z. angew. Chem.* 18, I, 1528-1531(1905)) for the detn. of S in petroleum oils, which involves preliminary treatment of the oil with Br-satd. HNO₃ (concd.), followed by ignition with Na₂CO₃ and NaNO₃. A useful graph for finding the S corresponding to given wts. of BaSO₄ is included. This graph also corrects for the amt. of salts occluded with the ppt. Details of the procedure are given and sources of error are considered.

F. W. PADGETT

Gasoline losses due to incomplete combustion in motor vehicles. A. C. FRIEDNER. A. A. STRAUB AND G. W. JONES. *J. Ind. Eng. Chem.* 13, 51-8(1921).—This investigation was undertaken primarily in connection with the ventilation of the proposed vehicular tunnel under the Hudson River. An exhaust-gas sampling app. and a gasoline-measuring app. to be connected directly to the carburetor, are illustrated and described. Av. results of tests under winter conditions are illustrated graphically. The content of CO for the majority of cars lies between 5 and 9%, while the av. CO in the exhaust gases for 23 cars tested was 6.7%, which is practically the ratio for develop-

ing max. power. The combustible gas in the av. automobile exhaust for one gallon of gasoline amounts to 30% of the total heat in a gallon of gasoline. Discussion by G. G. Brown and R. E. Wilson.

F. W. PADGETT

Process and equipment for refining benzene hydrocarbons. A. THAU. *Chem. Met. Eng.* 23, 1065-71 (1920).—Practice in Sweden and other parts of Europe is described. In the majority of large plants recently built, the light oil is fractionated according to h. p., the products are collected separately and each fraction is washed and redistd. after sufficient has accumulated, the process thus being intermittent (batch). In plants of another type, continuous running gives two streams, one containing about 90% C_6H_6 , and a smaller stream of higher-boiling hydrocarbons, while the residue is continuously run off to crystg. pans. This method requires only two storage tanks, and washing is simplified, but there is a slight disadvantage in that the process does not permit the sale of crude C_6H_6 , C_7H_8 , C_8H_{10} and solvent naphtha. Crude C_6H_6 and C_7H_8 give no trouble in washing with H_2SO_4 , as they sep. easily and do not generate much heat. But crude solvent naphtha reacts vigorously with both acid and caustic lye, and forms emulsions which entrain much valuable oil, up to 30-40%, and may not settle for several days. Hence original sepn. into two fractions only is an advantage, as the cut containing the solvent naphtha is dild. with about $\frac{1}{3}$ of C_7H_8 , etc., and can be handled much better than solvent naphtha alone. However, more care is necessary in the subsequent final distn. The acid ppts. most impurities, forming a tar, which is fluid while warm, but may become solid on cooling; heavier hydrocarbons are polymerized and remain in soln. They are recovered as residues in redistn., and may be coned, to any desired degree *in vacuo*, forming coumarone resin. For neutralization, NaOH soln. (d. 1.15) is best; Na_2CO_3 and $Ca(OH)_2$ may also be used. Preliminary washing with H_2O to remove suspended acid is customary. The re-use of used acid is not economical, on account of settling of impurities in lines and tanks. Acid should be regenerated after the first use. The washers are elevated, so as to permit running out by gravity. They usually have the form of vertical cylinders, of such size that they can be completely emptied into the stills. The material may be cast Fe 1.5-2 in. thick, or Pb-lined wrought Fe. The difference in wt. between the two types of construction is not great. The rule has been to use hars cast Fe 1.75 in. thick, which is kept in operation until leaks develop, then holes are cut in the shell corresponding to the places eaten away, Pb run in to fill the holes, and a lining of Pb 0.5-0.625 in. thick applied, being fastened to the Pb plugs. It is still cheaper to calk the leaks with soft Pb, and after too many repairs become necessary, replace the washer. Wrought Fe washers should be welded at the seams, and given a homogeneous lining of Pb 0.5 in. thick. One type of washer for both cast and wrought Fe consists of sections with horizontal and vertical flanges, the Pb lining also being in sections and bolted between the flanges. But this form has the disadvantage of a large no. of lodging places for acid and impurities. The cover of the washer has one manhole for inspection, and openings for acid, caustic, and hydrocarbon lines. The bottom is usually in the shape of a cone or hemisphere, the former being better for rapid and sharp settling. The acid and oil are agitated by a propeller or turbine at the bottom, to force the acid up through the hydrocarbons, or it may work downwards to force the hydrocarbons through the acid. The propeller is often provided with a jacket in the form of a cylinder or frustum of a cone, to aid in circulation. The most modern forms have a long narrow cylindrical stirrer, flaring at the bottom to a trumpet-like turbine, with many fine openings in the periphery, and slots on the face. The hydrocarbons pass down the tube and are hurled out at the bottom. Another form, for full charges in the washer, has the turbine at the top. Blowing with air is not practicable, on account of the vaporization, but ~~where~~ air connections are necessary for ventilation, blowing out, etc. Another type of washer circulates the acid and oil through a centrifugal pump, spraying the mixt.

in at the top through a perforated pipe. No type of continuous washer has yet given promising results.

M. R. SCHMIDT

BATTLE, JOHN ROME: *The Handbook of Industrial Oil Engineering*. Philadelphia: J. B. Lippincott Co. 1065 pp. \$10. For review see *J. Frank. Inst.* **191**, 140(1921).

SAUER, A., GRUBE, G., BURCHARD, E. VON DER AND SCHMIDT, OSKAR: *Die Verwertung des Ölschiefers*. Stuttgart: Konrad Wittwer. 31 pp. M. 3.50. For review see *Tonind.-Ztg.* **44**, 1295(1920).

Recovering sludge acid, etc. P. W. WEBSTER and V. K. BOYNTON. U. S. 1,361,940, Dec. 14. Sludge acid from petroleum refining is placed in a chamber with a porous bottom which may be formed of "filtros" or similar material and heated air or gas is forced through the bottom of the chamber into the acid. The very minute bubbles of gas rise slowly through the liquid with but little tendency to coalesce. The gas may be supplied at a temp. above 500° and effects concn. of the acid by carrying off the H₂O at lower temps. than are ordinarily required to effect vaporization of H₂O from H₂SO₄. Conservation of the acid and economy of heat in concn. are thus secured. Hydrocarbons of the sludge such as usually tend to form tarry deposits are largely oxidized by this process and the satd. hydrocarbons thus formed are distd. Granular C forms in the acid at the same time. A similar method may be adopted for the concn. of ordinary dil. H₂SO₄ or evapn. of H₂O or other liquids generally, at temps. below those ordinarily required.

Bituminous compositions. BARBER ASPHALT PAVING CO. Brit. 151,639, Sept. 27, 1920. Crushed quartz, granite, trap, or other igneous rock, sand, or clay composed of particles varying in size from dust to grit, is incorporated with melted asphaltum free from calcareous substances to form an anti-corrosive compn. for lining tanks. In an example, 15-35 parts of asphaltum, 25-30 parts of rock, etc., not coarser than 1/16 in. and 30-45 parts of rock, etc., between 1/16 in. to 1/4 in. are used.

Apparatus for recovering volatilizable material from shale, etc. G. A. BRONDER. U. S. 1,361,005, Dec. 7. Heated air and vapors are passed through the material to effect distn.

Recovering crude oil from "B. S." I. S. JOSEPH. U. S. 1,362,105, Dec. 14. The pat. relates to centrifugal sepn. of crude oil from "B. S."

Gasoline filter. A. J. FUSON. U. S. 1,361,243, Dec. 7.

Apparatus for producing gasoline by cracking heavier hydrocarbons. F. E. WELLMAN. U. S. 1,362,160, Dec. 14.

23—CELLULOSE AND PAPER

A. D. LITTLE

Cryptogamous and aquatic plants: their suitability for paper making. CH. GROUP. *Papier* **23**, 259-63(1920); cf. *C. A.* **14**, 2985.—Various cryptogamous and aquatic plants are described from the point of view of their utilization for paper making. The Pisan marshes contain a very large variety of plants, the ensemble of which is known locally as "falasco." Part of it is used for the manuf. of mats, cordage, baskets, wicker work, etc. Expts. were carried out on several of the species to det. the yields of pulp. The plants were cut, cooked with 6% NaOH under 3 atm. pressure for 3 hrs., washed, bleached with 10% of bleaching powder, pressed and dried. The following yields were obtained: *Carex paludosa* 43%; *Phragmites communis* 47.5%; *Scirpus lacustris* 45.5%; *Thypha angustifolia* 44%; *Cyperus longus* 25%. The bleaching of the fibers by SO₂ and by KMnO₄ was no more satisfactory than with bleaching powder. The

species which predominate in "falasco" yield long regular fibers, which could be easily utilized.

A. P.-C.

Determination of the degree of beating of paper pulp. ANON. *Papier* 23, 255-6 (1920).—The Schopper-Riegler app. for detg. the degree of beating of pulp and the method of carrying out the test are described.

A. P.-C.

Measuring the translucency and opacity of paper. RAYMOND FOURNIER. *Papier* 23, 257-9 (1920).—In Klemm's app. for measuring the opacity of paper, the personal equation plays a very great part. F. proposes a modification of this app. wherein the eye of the observer is replaced by a Se plate (similar to those used in telephotography), which is connected to a source of elec. current and to a galvanometer. By closing the circuit and noting the deflection of the galvanometer needle, first when there is no paper between the source of light and the Se plate, and, second, when one or more sheets of the paper to be tested are interposed, the opacity of the latter can be easily, quickly and accurately detd.

A. P.-C.

The manufacture of corrugated paper board. A. LAMBRETTIE. *Papeterie* 42, 1014-8, 1058-65 (1920).—The equipment used for the manuf. of corrugated paper board is described.

A. P.-C.

Sizes (for paper) (Brit. pat. 150,280) 14. Aluminium compounds for sizing paper (Ger. pat. 319,420) 18. Lignin and its reactions (KLASON) 10.

BROMLEY, HENRY A.: *Paper and its Constituents*. London: E. & F. N. Spon Ltd. 228 pp. 16. For review see *Can. Chem. J.* 4, 347 (1920).

Recovering values from waste sulfite liquor. R. W. STREHLENER. U. S., 1,361,506, Dec. 7. Acid gases or vapors (such as "blow-off" digester gas containing SO_2) are introduced into an autoclave containing waste sulfite liquor from cellulose manuf. and the temp. and pressure are sufficiently raised to convert SO_2 present into H_2SO_4 with formation of free S. Gas from the autoclave containing free S is then led to a container where it is oxidized with air to produce SO_2 for continuation of the process.

Treating wood-pulp liquor. B. GRATZ. Brit. 150,571, Nov. 26, 1919. Wood-pulp liquor, the residual lye from the sulfite cellulose process, is de-acidified and rendered suitable for the prepn. of artificial stone, by treatment with the ashes of fuel, particularly the ashes of lignite or other softer and brownish coals of comparatively recent vegetable origin. The liquor and ashes are moved relatively on the counter-low principle in an inclined trough. A suitable construction is specified.

Pyroxylin composition. J. M. KESSLER. U. S. 1,360,750, Nov. 30. Pyroxylin 100, denatured alc. 60, and acetin (free from monoacetin and containing about 65% riacetin) 28 parts are mixed and kneaded at a temp. of 40-55° until colloided and then rolled and formed into sheets or other desired forms. Stabilizers, coloring agents and auxiliary solvents may be used in addition to the ingredients specified. The use of an acetin free from monoacetin obviates the difficulties encountered from the hygroscopicity of the latter and facilitates the production of a transparent film. A suitable acetin for use in the process may be prepd. by treating glycerol with 5-6 mol. proportions of glacial HOAc , using 0.1% H_2SO_4 as a catalyzer and distg. off dil. HOAc resulting from the reaction until the distillate coming over reaches a concn. of 90% HOAc , neutralizing the H_2SO_4 with NaOAc and distg. off excess HOAc first at ordinary pressure and then *in vacuo*. This produces a neutral product containing a high proportion of riacetin. Liquid coating compns. may be produced by employing a larger proportion of volatile solvent than specified in the above-described mixt.

Paper pulp from resinous wood. R. H. MCKERR and A. A. HOLMES. U. S. 380,668, Nov. 30. Resinous wood is subjected to the action of liquid SO_2 in order to

remove resins and is then converted into paper pulp by the acid sulfite method or other suitable operation.

Paper or felted sheets. L. KIRSCHBRAUN. Brit. 151,029, Mar. 6, 1919. Relates to the manuf. of a waterproof paper or felted sheet, the object being to combine with the fibrous material a waterproof binding material, preferably a bituminous product such as asphalt. The bitumen, heated, if necessary, to make it fluid, is mixed by violent agitation with an emulsion of H_2O and clay heated to a temp. of from 300–400° F., the bitumen being added gradually to the paste or emulsion and more H_2O added as required. In place of asphalt, coal tar pitch, linseed or other oils may be used, and a suitable filler such as sawdust or cork may be added. This "matrix" is then mixed with fibers or paper stock in the beating engine, etc., and made into paper on a fibrous sheet in the usual way. A satisfactory matrix consists of 50 pts. of asphalt, 10 of clay, and 40 of H_2O , while the finished product may consist of 55–60 pts. of asphalt, 11–12 of clay, and 34–38 of fiber. The product is adapted for use as waterproof paper, for roofing and floor coverings, shipping cases, the counters, heels, toes, and inner soles of boots, etc., receptacles such as tanks, pails, etc., elec. insulation, etc.

Loading and coating paper, cardboard, etc. D. MOTTA, E. RUGIU and A. VILLAIN. Brit. 151,381, June 27, 1919. A fire-resisting and practically incombustible material having plastic properties is formed by mixing paper paste with an equal wt. of a mixt. of equal parts of $CaCO_3$ and $MgSiO_3$. The product may be used as a substitute for plaster compns., gelatin, paste, and other substances in the production of stereotype materials and the like and of walls which may be rendered damp-proof by varnishing.

24—EXPLOSIVES AND EXPLOSIONS

CHARLES E. MUNROE

Modern commercial explosives and their uses. A. J. STRANE. *Trans. Am. Inst. Mining Met. Eng.* 1920, 7 pp. (advance copy).—S. describes nitroglycerin, blasting gelatin, straight nitroglycerin dynamites, straight gelatin dynamite, extra, or ammonia, dynamites and gelatin, low-freezing nitroglycerin explosives, permissible explosives, non-freezing explosives and black blasting powder, giving in general the compn., properties and form of commercial packages. Blasting gelatin composed of 92 parts nitroglycerin and 8 parts guncotton is the standard for 100% explosive. Straight nitroglycerin dynamites are put up in 14 different strengths according to the nitroglycerin content running from 15 to 75% strength. Other high explosives in the U. S. are compared on a strength basis with these, placing by comparison NH_4 picrate at 33%, TNT 52.6, picric acid 66, and tetryl 76.8%. Since commercial nitroglycerin may freeze at 10°, a temp. frequently encountered where dynamite is used, and frozen dynamite is dangerous and difficult to use, the freezing point in dynamites is reduced by admixture of other substances. At first mononitro aromatic compds. were used, then more highly nitrated ones, and latterly dinitrochlorohydrin, tetranitrodiglycerol, dinitroethylene glycol and dinitropropylene glycol. In instances these last mentioned compds. are substituted for the nitroglycerin in dynamites.

CHARLES E. MUNROE

Engineering problems in dust explosion prevention. DAVID J. PRICE. *Chem. Met. Eng.* 24, 29–32 (1921).—This paper deals with the causes and factors affecting dust explosions covering the types of industrial plants, the ignition temps. of gases and dusts, the velocity of flame in dust air mixts., the pressures developed in dust explosions, and the relation of humidity to explosion frequency; data relating to these, assembled from the literature, are presented. The pressures developed by the explosion of grain dust-air mixts. are greater than those of coal dust-air mixts. Thus a mixt.

containing yellow corn dust generated a pressure of 15.2 lbs. per sq. in. as compared with 10.1 for Pittsburgh coal dust. Expts. are in progress for detg. necessary proportions of dusts in mixts. with air to produce explosion as has already been done for gases.

CHARLES E. MUNROE

The ignition of gases at reduced pressures by transient arcs. W. M. THORNTON. *Phil. Mag.* 40, 450-61(1920).—The nature of transient arcs is discussed. The results with direct current and with alternating current break sparks are shown. Two phases of ignition consisting of ionization and thermal effect are recognized. Their combined effect results in a sudden change in the condition for ignition. The effectiveness of thermal action is favored by higher gas pressure, whereas that of elec. action is favored by lower; these two opposing influences lead to reversal and oscillative effects in the current-compn. curves for ignition by direct current arc. Various curves and formulas are presented for which the original must be consulted. H occupies a unique position among the combustible gases, which is approached, however, by the higher members of the CH_4 series. The ignition of CO and coal gas is compared for d. c. and a. c. The difference for d. c. is slight, but for a. c. coal gas is far easier to ignite, owing to the greater sensitiveness of H than of CO.

S. C. LIND

Preparation of pure carbon dioxide (testing stability of gun cotton) (FARMER) 1. Electrical purification of fumes and gases in explosives manufacture. (DELAFFALLE) 4. Behavior of mine gases at high temperatures (WINTER) 21. Nitration process (Can. pat. 206,337) 10.

Die chemische Untersuchung der Grubenwetter. Kurzgefasste Anleitung zur Ausführung von Wetteranalysen nach einfachen Methoden. 3rd Ed. Edited by Otto Brunk. Freiberg: Cray and Gerlach. For review see *Z. angew. Chem.* 33, II, 416(1920).

Explosives. WENDELL R. SWINT. Can. 206,330, Dec. 7, 1920. An explosive contains gun cotton of high nitration 40%, liquid nitroxylenes 25% and NaNO_3 35%. Gun cotton of a lower degree of nitration may be used in certain proportions.

Explosives. LEON O. BRYAN and W. R. SWINT. Can. 206,329, Dec. 7, 1920. An explosive contains nitroglycerin 5%, trinitrotoluene 40% and NaNO_3 55%.

Explosives. A. VOIGT. Can. 206,392, Dec. 2, 1920. Salts of the sulfo acids of nitroresol are thoroughly mixed in suitable proportions with nitrates, chlorates and perchlorates of Na.

Explosive. R. L. HILL. U. S. 1,360,397, Nov. 30. Explosives adapted for use in blasting are formed of NH_4ClO_4 45-55, nitrostarch 35-45 and an oil or oily nitro compd. such as nitrated solvent naphtha 0-5 parts. U. S. 1,360,398-9 relate also to explosives containing NH_4ClO_4 45-55 and nitrostarch 35-45 parts, with or without ingredients such as oils, nitrates, S or MnO_2 , which may be added for the purpose of modifying the explosives to adapt their properties to various requirements. Cf. C. A. 14, 1609.

Explosives. E. I. DU PONT DE NEMOURS & Co. Brit. 150,299, May 19, 1920. Consists in avoiding or removing metallic fouling in gun barrels by incorporating metals or alloys in the propellant explosive itself instead of adding them to the propellant charge or cartridge. E. g., 2-5% of powdered Sn is added to a nitrocellulose explosive while it is being treated with solvent. An alloy of Sn and Pb with or without Zn may be used instead of Sn. Other usual ingredients such as diphenylamine, nitroglycerin, graphite, and nitro compds. may be added to the explosive.

Blasting detonators. W. ESCHBACH. Brit. 151,572, May 28, 1920. Consists in making the casings of such detonators of Al when the charge does not contain ful-

minate of Hg, but contains compns. inert toward Al, *e. g.*, tetranitromethylaniline with a top layer of PbN_3 .

Blasting cap charge. CHAS. M. STINE. Can. 206,311, Dec. 7, 1920. A compn. used in charges for blasting caps for primers in the detonation of high explosives consist of an org. compd. having a nitro group and nitrate group and a priming material $\text{C}_6\text{H}_5(\text{CH}_2\text{NO}_2)_2(\text{NO}_2)_2$, $\text{C}_6\text{H}(\text{OH})(\text{CH}_2\text{NO}_2)_2(\text{NO}_2)_2$, $\text{C}_6\text{H}_2(\text{CH}_2\text{NO}_2)_2\text{NO}_2$ or $\text{C}_6\text{H}(\text{CH}_3)(\text{CH}_2\text{NO}_2)_2(\text{NO}_2)_2$ may be used with a priming charge of Hg fulminate.

Incendiary composition. G. W. WEBB. Can. 206,374, Dec. 7, 1920. The compn. comprizes granulated Mg, a nitrate and MgO formed into a coherent mass by compression.

Match-head composition. W. A. FAIRBURN. U. S. 1,360,282, Nov. 30. A match-head compn. which is not deleteriously hygroscopic is formed of NaClO_2 , 20, Zn dust 5, a binder such as glue 7.5, combustible material such as P 3.75, and a filler such as BaCO_3 , CaCO_3 or CaSO_4 25 parts, with or without additional oxidizing salts.

Match-head composition. W. A. FAIRBURN. U. S. 1,360,283, Nov. 30. Fe_2O_3 is used in match-head compns. together with a metal such as Al or Mg and Si whose normal oxide has a greater heat of formation than has Fe_2O_3 , in order to give a high temp. on ignition. KClO_4 and usual combustible, binding or filler ingredients also are used in the compn.

25—DYES AND TEXTILE CHEMISTRY

L. A. OLNEY

Dyeing of artificial silk. AUG. PETERS. *Deut. Färber-Ztg.* 56, 901-2(1920).—The manuf. and chem. nature of artificial silks, dyeing with substantive dyes, dyeing with basic dyes, bleaching and finishing are described with much detail. L. W. R.

"Pinking" of bleached cotton goods. W. B. NANSON. *Cotton* 85, 19-23(1920).—The writer considers, at length, the causes of "pinking" in bleached cotton goods, especially the canvas used in white canvas shoes, methods of avoiding this, and of restoring the clear white color to goods already discolored. It is believed that this bluish pink coloration developing upon shoe canvas is caused primarily by the blueing material used at the bleachery, and that this treatment is unnecessary, as the subsequent steaming and pasting to which the material is subjected at the shoe factory, causes an oxidation, converting the blueing largely into ferric hydrate and ferric chloride, producing a dirty brownish yellow stain. In the shoe industry, the most probable cause of "pinking" is believed to be due to the fumes of aniline and toluidine, arising from the "tapsticker" used in putting on the soles of shoes, or where the soles are of rubber, from the soles themselves, but this has been found only where the canvas has been blueed with Prussian blue. NATHAN VAN PATTEN

Finishing drills for shoe linings. W. B. NANSON. *Cotton* 85, 92-5(1920).—The use of starch and glucose for stiffening shoe linings and soaps for softening is discussed. Formulas and methods of application are given. NATHAN VAN PATTEN

GILL, A. H.: Use of Oils in Textile Mills. Boston: "Textiles." 54 pp. For review see *Manufactures Record* 79, No. 2, 132(1921).

PANIZZON, GIACOMO: Trattato di chimica delle sostanze coloranti artificiali e naturali. Part II, Section I. 432 pp. L. 38. Section II. 468 pp. L. 38. Milan: Ulrico Hoepli. For review see *Boll. chim. farm.* 59, 460(1920).

TRUTWIN, H.: Enzyklopädie der Küpenfarbstoffe, ihre Literatur, Darstellungsweise, Zusammensetzung, Eigenschaften in Substanz und auf der Faser. Berlin: J. Springer. 868 pp. M. 130.

Dyes. W. CLARK. Brit. 151,657, May 8, 1918. Coloring matters sol. in fats and oils are obtained by treating basic dyes with naphthenic acids or montanic acid in the presence of $Al_2(SO_4)_3$. Examples are given of the treatment of methyl violet, malachite green, and alizarin. The products can be used for printing purposes, or for coloring resins, varnishes, lakes, waxes, oils, fats, etc. Cf. 2,878, 1886, 7,635, 1891, and 12,095, 1912 (C. A. 7, 3667).

Dyes. L. CASSELLA & Co., GRS. Brit. 151,000, Aug. 27, 1920. Sulfuretted dyes, which are probably thiazines, are obtained by sulfurizing β -hydroxynaphthoquinonearylamides ($O:OH:NR = 1,2,4$); suitable sulfurizing agents are S, alone or in presence of catalysts, solvents, or diluents, disulfur dichloride, S_2O_4 , or alkali polysulfides, preferably in alc. soln. at temps. not exceeding 150° . The resulting products, if they contain sulfonic or carboxylic acid groups are acid mordant dyes, or if they are free from such groups are vat dyes; the vat dycings may be after-treated with metal salts, preferably Cr salts, giving fast green to black shades. Numerous examples are cited.

Dyeing; dyes. FARBWERKE VORM. MEISTER, LUCIUS & BRUNING. Brit. 150,329, Aug. 20, 1920. Fast shades are obtained by after-treating with HCHO, or substances yielding it, cotton or similar fibers dyed with azo dyes containing as end components one or more mols. of aminoarylpyrazolones (except the dyes described in German Specification 289,350 (C. A. 10, 2802)); the treatment with HCHO is preferably effected in hot acid soln. According to examples, cotton is dyed with (1) the secondary disazo dye aniline-*p*-aminobenzoyl-2,5,7-aminonaphtholsulfonic acid—1-*p*-aminophenyl-3-methyl-5-pyrazolone; (2) the disazo dye from tetrazotized benzidine-3,3'-disulfonic acid and two mols. of 1-*p*-aminophenyl-3-methyl-5-pyrazolone; (3) the disazo dye from tetrazotized *m,m'*-dichlorobenzidine and two mols. of 1-*m*-aminophenyl-5-pyrazolone-3-carboxylic acid, and in each case after-treated with HCHO in hot HOAc soln., orange, brownish orange, and reddish yellow shades, resp., being obtained.

Secondary diazo dyes for wool. SOC. ANON. POUR L'IND. CHIM. A BALZ. Ger. 325,062, Apr. 11, 1916. The tetrazo compd. of 3,3'-diamino-4,4'-dimethyldiphenylmethane is combined with 2 mols. of the same or different hydroxynaphthalenesulfonic acids or with 1 mol. of a hydroxynaphthalenesulfonic acid and 1 mol. of another suitable coupling component.

Dyes; intermediate products. L. CASSELLA & Co., GRS. Brit. 150,709, Sept. 1, 1920. Addition to 148,339 (C. A. 15, 441). The vat dye described in the principal patent is obtained by reaction of hydrazine on anthraquinone-1,2-isoxazole; the parent isoxazole is prepd. by treating 1-nitro-2-methylantraquinone with fuming H_2SO_4 .

Dyes; dyeing; intermediate products. P. A. NEWTON. Brit. 3,557, 1914. Monazo and disazo dyes are obtained by combining with aminoarylpyrazolones the tetrazo compds. of the aminobenzoylamino compds. described in 1,441, 1911 (C. A. 6, 1853), and 18,205, 1911 (C. A. 7, 420), or the monodiaz compds. of aminobenzoylaminothiazolesulfonic acids. The products dye cotton yellow shades, which are rendered fast by after-treatment with HCHO, or can be diazotized on the fiber and developed, e. g., with β -naphthol or 1-phenyl-3-methyl-5-pyrazolone. Examples are given.

Dyeing. SOC. CHIMIQUE DES USINES DU RHÔNE, ANCIENNEMENT GILLARD, P. MONNET, ET CARTIER. Brit. 150,989, May 17, 1920. To permit the dyeing of cellulose acetate by ordinary processes, the threads, films, etc., having this material as basis are treated with hot or cold solns. of metallic salts, to which alkali has been added. A soln. of NaCl and NaOH is given as an example of the soln. employed. Cf. 20,872.

Dyeing and preserving textile fabrics. O. L. COLE. U. S. 1,361,139, Dec. 7. Canvas, yarn, or similar textile fabrics are treated with a soln. containing tannic acid and the fabric is subsequently immersed in a bath formed by suspending $CaCO_3$ in a

soln. of CuSO_4 in order to protect the material from mildew or other destructive parasites.

Simultaneous breaking up and dyeing of bast material. R. HALLER. Ger. 318,271, Jan. 25, 1919. Reducible dyes, added to the alk. disintegrating bath, dye the opened bast fibers, acting in conjunction with pectin compds. derived from the plant substance forming during the boiling process and having a reducing action in alk. medium. Na_2S or other alk. sulfide may be added to the disintegrating bath. The sepd. fibers are obtained in the desired color. Disintegration and dyeing are effected in a single process, and the color is claimed to be very fast.

Dye soap and mordant. D. J. BLOCK. U. S. 1,361,811, Dec. 14. A dye soap in flake form is mixed with a mordant such as Na_2SO_4 and Na_2CO_3 or tannin.

Printed fabrics. SOC. D'IMPRESSION DES VOSGES ET DE NORMANDIE. Brit. 151,286, Sept. 20, 1920. The appearance of moiré or iridescent wool or silk is obtained on a cotton fabric by first weaving a "grogan" or "granite" or "imitation grogan" effect or pattern on a cotton fabric by means of a jacquard or dobby, or, alternatively, by printing such an effect on a plain fabric, and then printing on the whole or portions of one or both sides of the fabric a fine-ribbed stripe or "milleriaies" effect. Further decorative patterns, etc., may be printed on the fabric.

Printing fabrics. CALICO PRINTERS' ASSOCIATION. Brit. 151,056, June 5, 1919. Half-discharge effects on colored grounds are obtained by first printing cotton fabrics with thickened solns. of alkali sulfites, with or without metallic mordants, then printing, padding, or otherwise impregnating the fabric with metallic mordants, fixing, darning, and then either dyeing with alizarin or other dyes appropriate to the mordant or tanning and dyeing with basic or other appropriate dyes. The fabric may be unprepd. or may be previously treated with a turkey-red oil, β -naphthol, or chlorate prepare. Citric acid or lime-juice resists may be printed on simultaneously with the sulfite half-resist. Numerous examples are cited.

Printing textiles. FARBWERKE VORM. MEISTER, LUCIUS & BRUNING. Brit. 150,303, July 14, 1920. Addition to 147,102 (C. A. 14, 3800). C_2H_4 thiodiglycol is employed as a constituent of the printing paste in printing with H_2O -sol. org. dyestuffs. It may be employed, e. g., in printing with cerulein, alizarin red, alizarin blue, acid alizarin black, galloxyaniline, dianil chrome brown G, thiogene cyanine BB extra, the azo dye from diazotized 2-aminoanthraquinone-3-sulfonic acid and acetoacetanilide, fulling scarlet 4R conc., etc., and in cases where a grounding of naphthol or naphthol A S soln. is used. Examples are given.

Treating vegetable fibers. SOC. GILLET ET FILS. Brit. 150,665, Dec. 12, 1919. Vegetable fibers of all kinds and in any stage of manuf. are treated by fixing thereon the products of the hydrolysis of proteic substances to impart to the fibers the character of wool, both physically as to feel, appearance, and calorific qualities, and chemically as regards their affinity and absorbent capacities for dyestuffs. The treatment of the fibers is also a prepn. for the usual printing processes. The fibers, whether in the mass or in the state of lap, cops, card ends, twistings, yarns, or fabrics are treated with the liquid obtained by the action of strong acids, alkalies, or other hydrolyzing agents on proteic substances to which oxidizing agents may be added, and are then washed with H_2O with, if desired, a preliminary washing with a dil. acid or alkali, or a saline soln. The fibers may finally be treated with HCHO , alone or with NH_3 , or with phosphoric acid. In a modification of the process, the fibers are impregnated with a protein soln., the deposit being treated, if desired, with HCHO and tannin. The fibers may then be dried and are subsequently treated with a hydrolyzing agent, with or without the addition of an oxidizing agent, and are finally washed with H_2O , after the preliminary washing if desired. Casein, egg albumin, serum albumin, and gelatin are

mentioned as suitable proteic substances, and HNO_3 , H_2SO_4 , HCl , H_3PO_4 , and formic acids, ZnCl_2 , and NaOH are given as examples of suitable hydrolyzing agents.

Retting flax. B. S. SUMMERS. U. S. 1,360,329, Nov. 30. During retting of flax, insol. products only are removed from the bath in order to prevent fouling of the liquor of a circulating bath.

Retting flax. L. A. JOHNSON. Brit. 151,143, Aug. 20, 1919. In obtaining fibers from flax, the straw, prior to retting, is treated in one stage, or in a number of stages extending over successively increasing periods of time, with an alk. solvent to remove the whole or a greater portion of the gum and resin compds., and the retting operation is carried out in an alk. medium. A natural soda, such as Magadi soda, occurring in East Africa, may be used, and in the retting operation, which is preferably effected at $80\text{--}95^\circ\text{F}$., the liquid is continuously aerated and circulated.

Removing stains from fabrics. A. S. CUSHMAN. U. S. 1,361,833, Dec. 14. Ink stains, grass stains and similar stains are removed from cloth or paper by the action of dil. KMnO_4 soln., followed by the application of an oxalic acid soln. with the addition of H_2O_2 and a subsequent washing with H_2O .

26—PAINTS, VARNISHES AND RESINS

A. H. SABIN

The detection of oils other than linseed in paints by means of the hexabromide number of the fatty acids. HERBERT BAILEY AND WALTER D. BALDSIEFEN. *J. Ind. Eng. Chem.* 12, 1189-94 (1920).—Exptl. work leading to modifications (see *C. A.* 14, 3804) of the Steele-Washburn method (*C. A.* 14, 356) for detg. hexabromides is given. Tabulated yields detd. by the authors' method on a large no. of linseed and other vegetable oils and on mixts. of these, show an av. of 42% for pure linseed, 6% for pure soy, 0% for tung and fish oils. Heat-treated linseed oils show low yields but commercial boiled oils give practically normal amts. of hexabromides. Fish oils give insol. octabromides recognized by their lack of a m. p. before decomposing, and their insol. in warm CHCl_3 . The proportion of linseed oil in mixts. with one of the above oils can be detd. much more closely from their hexabromide than from their I value. It appears probable that the method may give the approx. compn. of the non-volatile vehicle of a paint, but further study of the effects of other paint constituents is necessary.

F. A. WERTZ

The wrinkling of varnish films. HERMANN VOLLMANN. *Farben-Ztg.* 26, 58-8 (1920).—To study the effect of the solvent medium on the wrinkling of varnish films dried under various conditions of light (cf. *C. A.* 14, 356), V. prepd. varnishes from the same base using turps, solvent naphtha, tetralin extra, benzine (b. p. $128\text{--}76^\circ$), primary C_6H_6 distillate (b. p. 66°), and benzine (b. p. $65\text{--}94^\circ$). These were flowed onto glass plates and allowed to dry and age under colorless and under red glass, in the dark, and without free access of air. All the films except from varnishes contg. C_6H_6 and low h. p. benzine, remained practically free from wrinkles for 6 mos. under all conditions. The two exceptions were more wrinkled under white light than under the red light or in the dark, but remained perfectly smooth in the chamber where there was no free access of air. The use of highly volatile thinners is apparently conducive to wrinkle formation. The soly. of the varnish base in the solvent, ease of diffusion of the solvent vapors through the surface of the film, and their chem. or catalytic action on the remaining varnish base, as well as the nature of the driers, resins, and oils present, age of the varnish, etc., might all influence the results. Lack of free access of air might have prevented wrinkling through slower evapn. of the solvent, slower oxidation of the drying oils, higher humidity or temp. V. suggests a more thorough study of the subject.

The lac industry in India. ANON. *Drugs, Oils and Paints* 36, 200-1, 266-8 (1920).—A review of production and manuf. methods, etc. F. A. WERTZ

Factory control in the varnish industry. ERICH STOCK. *Farben-Ztg.* 26, 21-2 (1920).—S. calls attention to the necessity, not only for chem. control, but for a perpetual inventory of all raw materials; and suggests a system for the latter.

F. A. WERTZ

A contribution to the melting and cooking question. ANON. *Farben-Ztg.* 26, 25(1920).—The author points out some of the advantages of using jacketed kettles in which superheated H_2O is circulated.

F. A. WERTZ

Summary of bulking value tables, computed to American and British standards. HENRY A. GARDNER. *Circ.* 112, *Paint Manufs. Assoc. of U. S.*, 3 pp., Jan., 1921.—A summary of *Circ.* 104 (*C. A.* 14, 3161).

F. A. WERTZ

The manufacture of paints and varnishes for railroad coaches. OSKAR PRAGER. *Chem. Tech. Fabricant*, Nos. 8 and 9(1919); *Rev. chim. ind.* 28, 332-5(1919).—Exterior and interior paints for railroad coaches must withstand extreme conditions of cold, heat, fumes, smoke, dust, shocks, etc. They must protect iron from rust and wood from swelling, decaying, splitting, etc., and possess a pleasing appearance. To give durability and brilliance the paint may be covered with a varnish, but it is more economical to unite these two qualities in one product. The pigments must satisfy all special conditions, be resistant to the influences of water, light, oil, alkalies, acids, etc., possess good covering power, and be cheap. The pigments used are largely mineral products. Colors are produced by mixing a white or gray base such as lithopone, white lead or zinc oxide, with a suitable colored pigment such as zinc blend, graphite, mica, or carbon black for grays; ultramarine or Prussian blue for blues; ochers, chrome yellow, chrome orange, or sienna for yellows; green mineral or a mixt. of yellow and blue for greens; oxides of iron, chrome red or red lead for reds; and umbers for browns. The vehicle is polymerized linseed oil, wood oil, or a wood oil-congo resin varnish, possibly containing soy oil. A typical paint follows: 100 parts wood oil with 1000 parts resin are heated to 200-220°, 2 parts litharge, 1 part hydrated MnO_2 , then 5 parts zinc oxide and 5 parts lime are incorporated in small portions. The temp. is raised to 240-250°, 100 parts stand. oil (previously heated) are added and the mixt. is dild. with turpentine or benzine. This vehicle is mixed with pigment in an amt. proportional to the oil absorption of the pigment. Varnishes for railroad coach use contain various resins, preferably the hard varieties, wood or linseed oil, resinates, oleates or borates of Ph, Mn, Zn or Co, and thinners. The original article contains a description of the plant pigment essential to varnish making. The usual procedure is followed. Formulas as follows are included: Resin (Zanzibar, kauri or Borneo), ten parts; linseed varnish oil, 10 to 15 parts; stand. oil, 0 to 5 parts; Pb and Mn resinate, 0.5 to 1 part; lime 0.1 to 0.25 part; turpentine, 20 to 35 parts.

C. B. EDWARDS

Enameling in the automobile industry. M. R. ARMSTRONG. *Elec. J.* 18, 6-9 (1921).—The gas enameling ovens are being rapidly supplanted by elec.-heated ovens. Full details are given of the operation and regulation of two large elec. installations. The control of the ovens is entirely automatic. Temp. measurements, taken during the radiation test, show that for an av. oven temp. of 200° a temp. of 54° was maintained on the outside walls, with a room temp. of 19.5°. In another test 12 gallons of enamel were used on a daily output of 55 chassis. The trip through the oven required about 50 min. The av. hourly elec. consumption was 213 kw. hr. at 6 chassis per hr. Each chassis weighed 750 lbs.; or 21.1 lbs. per kw. hr.

C. G. F.

Electrolytic production of hydroxides from metals (pigments) (U. S. pat. 1,361,041)
Compositions and processes for coating glass surfaces (Brit. pat. 150,598) 19.

GARDNER, HENRY A.: *Papers on Paint and Varnish and the Materials Used in their Manufacture*. Washington, D. C.: P. H. Butler, 1845 B St., N. W. 501 pp. \$10. For review see *Paint, Oil Chem. Rev.* 70, No. 25, 16(1920).

Paints. M. R. ISAACS. Brit. 150,551, Oct. 8, 1919. A coating compn. consists of a protein substance such as casein, an alk. earth hydroxide such as CaO, a F compd. such as Na_2F_2 and filling or hinding materials such as Na silicate, silicates, aluminates, sulfates, etc., of Ca and Mg, plaster of Paris, asbestine, argillaceous materials such as clay, portland or other cement, and mineral or other pigments. A preservative such as naphthalene and a mineral, vegetable, or animal oil may be added.

Paint for ship bottoms or the like. H. BURSTIN. Ger. 319,199, Nov. 22, 1918. The paint consists of the normal or acid naphthbenic acid salts of Hg, Cu, As, Ni, Pb or other heavy metals, or of a mixt. of such salts. E. g., the alkali naphthenate soln., obtained from the petroleum products by means of lyes, is weakly acidified with dil. mineral acid, and the particular metal salt soln. is added until no more ppt. is formed. The acid naphthenate floats upon the aq. soln. as an oily viscous layer which can be drawn off or shaken out with an org. solvent. The resin or resins may be added up to 20% according to the hardness desired.

Converting lead sulfide, or ores containing the same, into sublimed white lead. G. m. b. C. SYNDICATE, LTD. Ger. 321,520, Apr. 6, 1916. The powdered PbS or ore is volatilized in an elec. furnace (preferably elec. arc.) at a temp. of at least 2760° ; the vapors are treated with gases containing O. The reaction product consists essentially of PbSO_4 , PbS and some Pb oxide and is obtained as an impalpable powder; it is sepd. from the gaseous medium in which it is suspended. When mixed with a drying oil, a paint is obtained. Preheated air is used in the process.

Antimony pigments. P. CHAILLAUX. Brit. 151,422, Aug. 1, 1919. Sb vermilion of the formula Sb_2S_3 and Sb_2S_4 are produced by passing a current of SO_2 or H_2S , or a mixt. of these two gases, into molten Sb_2S_3 . When SO_2 is employed, it may be prepd. *in situ* by incorporating S with the Sb_2S_3 and then injecting the requisite quantity of air or O into the mixt.

White pigment. P. FARUP. U. S. 1,360,737, Nov. 30. See Can. 201,704 (C. A. 14, 2722).

Tar paint. T. J. DUFFIN. U. S. 1,362,241, Dec. 14. A paint adapted for use on exposed metals or wood is formed of roofing tar and waste liquor from gas mains, mixed in equal amts.

Coumarone and indene resins from naphtha. S. P. MILLER. U. S. 1,360,665, Nov. 30. Resins of the coumarone-indene series are prepd. from naphtha by the action of AlCl_3 0.25-1.25% at temps. of 20° or lower. The polymerization may even be carried out at temps. as low as -20° . After the polymerization has been completed at a low temp., the mixt. is permitted to settle and the polymerizing agent is drawn off and the naphtha may then be washed with H_2O to remove more of the polymerizing agent. The remaining naphtha is then neutralized with an alkali, washed and sepd. The solvent naphtha may then be distd. in the usual way to remove the more volatile constituents, followed by steam distn. *in vacuo* to remove heavier oils. Higher yields of polymerization products are obtained in this process by reason of the low temp. maintained in the polymerization. By conducting the polymerization of the same naphtha at 10° instead of at 60° , the yield of resinous product was increased from 45% to 60% of the wt. of the naphtha treated. In general, the lower the temp. of polymerization the higher are the melting points and the lighter the color of the polymerization products formed.

Oxidizing oils. G.* SCHICHT AKT.-GES. Brit. 148,291, July 9, 1920. Addition to 147,661 (C. A. 14, 2806). Oils are oxidized by coating or impregnating with oil

the finely divided oxidized product, and exposing to air, O, etc. In an example 100 parts of linolin are mixed with 50 parts of linseed oil. The use of tungoxin is also specified.

27—FATS, FATTY OILS AND SOAPS

E. SCHERUBEL

The Baskerville process for the production of edible oils. CHAS. BASKERVILLE. *Cotton Oil Press* 4, No. 8, 43-6(1920).—Covers similar ground to article in *Chem. Age* (C. A. 15, 320) but in a more scientific manner.

H. S. BAILEY

The composition of rapeseed oil. CONRAD AMBERGER. Erlangen. *Z. Nahr.-Genussm.* 40, 192-201(1920).—The investigation was conducted to det. whether di-erucin, reported to have been found in rapeseed oil (*Korr. d. Vereins d. Bayer. Chem. Nr. 1; Ber. 19, 3320*(1896)), actually exists in the fresh oil as such or is a product of the hydrolysis of a triglyceride. Owing to the difficulty of distg. the glycerides from rapeseed oil without their destruction, hydrogenated oil was used. From hardened rapeseed oil A. isolated by repeated fractional crystn. from ether and CHCl_3 a triglyceride which was identified by its const. as *stearodibehenin* which could only have been derived from the triglyceride *oleodierucin* in the unhydrogenated oil. L. D. ELLIOTT

Coöperative soy-bean oil work. H. P. TREVITHICK. *Cotton Oil Press* 4, No. 9, 44-49(1921).—This is an outline of work on the color grading of soy-bean oils proposed for 1921 by the Am. Oil Chem. Soc.

H. S. BAILEY

Hydrogenation case adjudged. J. H. SHRADER. *Cotton Oil Press* 4, No. 9, 53-4(1921).—A digest of the U. S. Supreme Court's decision in the case of Berlin Mills Co. petitioner vs. Procter & Gamble Co. This final decision, reversing the lower courts, opens to all the manufacture of a food product by the partial hydrogenation of a vegetable oil.

H. S. BAILEY

Peanut-oil production in Marseille. A. V. DE FORD. *Cotton Oil Press* 4, No. 9, 54-5(1921).—In Marseilles the best grades of peanut oil are made from nuts imported in the shell largely from West Africa. Low grade oils are used mainly for soap making, but to some extent for lubricating and lighting. For making the edible peanut oil the nuts are always shelled and the red skins and germs removed by machinery before pressing. "Marseillaise" and Anglo-American hydraulic presses are both in use, but the first pressing is always made cold. The yields are 21-23% in the first pressing and 10-11% in the second. The cold-pressed oil is not refined, being merely filtered and if intended for margarine bleached.

H. S. BAILEY

Error in official method of sampling tank cars. P. W. TOMPKINS AND E. S. McELLIGORR. *Cotton Oil Press* 4, No. 9, 50-2(1921).—The official methods of the various oil assocns. for sampling tank cars do not specify that the sample be drawn from a well mixed car or immediately after loading. This may lead to gross errors, for if a uniform sample through the entire depth of a horizontal, cylindrical tank be taken (by trier), the sediment and water from the bottom form a larger portion of the sample than of the car. Specific examples are cited in which cars of peanut oil sampled after arrival at eastern points, showed 0.7% water when the sample drawn on the west coast as soon as the cars were loaded, analyzed only 0.4%. The free fatty acid in peanut and other meals is higher than in the oil from which these meals have settled out and the present sampling procedure, therefore, may also give too high free fatty acid figures.

H. S. BAILEY

History of vegetable oils (oleomargarine) 12. Calibration of sixteen Lovibond red glasses (PRIEST) 1. Priest photometer (AGGS) 1. Catalytic hydrogenation (Brit. pat. 150,991) 10. Dye soap and mordant (U. S. pat. 1,361,811) 25.

Hydrogenation of fats and oils. M. MATSUNAGA. Japan 35,928, Mar. 6, 1920. By passing through a red-hot tube, purified C_3H_8 is decomposed to H_2 and fine C. The gas thus produced is passed into fats and oils containing reduced Ni in hot state. Hydrogenation is successfully performed accompanied by deodorization and decolorization.

Saponaceous detergent. E. D. CHAPLIN. U. S. 1,362,393, Dec. 14. A solid detergent adapted for washing grease from the hands is formed of soap, H_2O , a small amt. of free alkali and granules of resin mixed with 20% of cotton fiber or similar material.

Soap from cereals. M. AISEN. U. S. 1,360,252, Nov. 30. A cereal such as corn, oats, wheat or rice is hydrolyzed by heating to a high temp., but below the carbonization point, which is about 180° for 2 hrs. This hydrolyzed product is then mixed with about 40% its wt. of Na_2CO_3 and the mixt. is sprayed with a soln. of NaOH, heated and treated with sufficient fatty acid to neutralize any free alkali present. The product thus obtained yields sol. very active surface colloids when employed with H_2O as a detergent.

Soap from cereals. M. AISEN. U. S. 1,360,253, Nov. 30. A cereal soap is prepd. by heating a cereal product such as corn, oats, wheat or rice to a high temp. for about 2 hrs. to effect hydrolyzation without carbonization, adding a fatty acid in the proportion of 1 part for each 1.75 parts hydrolyzed cereal and then adding a soln. of 1 part NaOH to each 5 parts cereal.

Cereal soap. M. AISEN. U. S. 1,360,254, Nov. 30. A cereal flour such as corn, oats, wheat or rice, is hydrolyzed by heating with a mineral acid, saponified with alkali and any excess alkali present is neutralized with fatty acid to produce a detergent.

Bleaching of soap from pupa oil. K. TAKANO. Japan 35,920, Mar. 4, 1920.

Crude soap is bleached by boiling with about 0.4% NaOH, 0.015% hyposulfite and H_2O .

28—SUGAR, STARCH AND GUMS

F. W. ZERRBAN

The difference between the direct polarization and the sucrose Clerget in (beet) molasses. E. SAILLARD. *Circ. hebdom. synd. fabr. sucre* 32, 222; *Deut. Zuckerind.* 45, 598(1920).—In dry yrs., when beets and molasses are high in N, this difference ranges from 0 to 0.88%, av. 0.53%, while in normal yrs. it is 1 to 2%, av. 1.45%. This difference is caused by the presence of nitrogenous substances which are levorotatory in alk., and dextrorotatory in acid soln.

F. W. ZERRBAN

A note on the determination of diastatic activity. EDWARD IVAN ROSENBLUM. *J. Soc. Chem. Ind.* 39, 311-3T(1920).—The activity of diastase is considerably affected by the H-ion concn. of the malt ext. and starch used. The p_H of both of these varies greatly, owing probably to small but varying amts. of phosphates. Using the method of Harrison and Gair (*Pharm. J.* 94, 6(1906)), and the indicator method of p_H detn., one malt ext. was found to exhibit its max. effect in the region of methyl red. With diastase purified by Wroblewski's method, and with sol. starch, the highest activity was found at a reaction slightly acid to methyl red and alk. to methyl orange, produced by addition of $NH_4H_2PO_4$. The diastatic effect increases up to a concn. of 0.5% of this salt and then decreases again. When using 0.5% of this salt detns. made with different starches give about the same result, which is otherwise not the case.

F. W. ZERRBAN

Purity of fourth mill juice as compared to that of bagasse juice from fourth mill. B. K. RUTHER. *Arch. Schweizerind.* 28, 1860-5(1920).—Reply to Böhrling (C. A. 15,

325).—The souring of the bagasse juice suspected by B. cannot be made responsible for the negative values for the colloidal water. If the bagasse juice does sour during the short time required for extg. it in the bagasse press, then the latter is useless and should be abolished. F. W. ZERBAN

Factory troubles. E. SAILLARD. *Circ. heb. synd. fabr. sucre* 32, 365; *Deut. Zuckerind.* 45, 662(1920).—During the 1920 campaign it was sometimes noticed that the alkalinity of the juice after the second satn. diminished gradually and finally disappeared entirely. This trouble is due to the activity of microorganisms and not to any chemical causes. It can be overcome by thorough mechanical cleaning of tanks and presses, and by washing the presses with a soln. of 0.5 l. formalin per 100 l. of water. Chloride of lime may also be used. Sterilization by heating to 120–125° for 15–20 min. is likewise effective. Even running very hot water through the freshly dressed presses helps to some extent. F. W. ZERBAN

Entrainment losses. W. C. NIEBOER. *Arch. Suikerind.* 28, 1889–91(1920).—N. confirms the results of De Haan (*C. A.* 14, 2274) and states that the installation of improved catch-alls has reduced the undetd. losses in one factory from 4.07% in 1918–9 to 2.65% in 1919–20. F. W. ZERBAN

Centrifugal pumps. BERTHOLD BLOCK. *Deut. Zuckerind.* 45, 587(1920).—The advantages and disadvantages of these pumps under sugar factory conditions are pointed out, as compared to piston pumps, and it is shown where and how they may be of greatest service. F. W. ZERBAN

Electric oven for rapid moisture tests (in raw sugars) (SPENCER) I.

Die Runkelrüben-Zucker-Fabrikation. Edited by the Verein der deutschen Zucker-industrie. Berlin: Paul Parey.

SORNAY, P. DE: *La canne à sucre à Pile Maurice*. Paris: Augustin Challamel. 677 pp. 50 fr. For review see *J. fabr. sucre* 61, No. 44, 2(1920).

WULFF, L.: *Beiträge zur Zuckerkristallisation*. Parchim, Germany: Kommissionsverlag H. Webdemann. M. 1. For review see *Intern. Sugar J.* 22, 648(1920).

Purifying beet molasses. W. D. BONNER. U. S. 1,362,078, Dec. 14. Beet molasses to be purified is dild. and treated with sufficient tartaric acid to ppt. the bulk of the K as acid K tartrate. The ppt. is sepd. and further purification is effected by evapn.

29—LEATHER AND GLUE

ALLEN ROGERS

Analysis of sole leather; the determination of water-soluble matter by the French and English methods. E. JALADE. *Le cuir* 8, 394–7(1919).—In the French method 5 g. degreased leather are shaken with 250 cc. water at 15° for 5 hrs., the soln. is allowed to stand 1 hr. and is then filtered with suction through a Berkefeld candle. An aliquot is evapd. to const. wt. In the English method 25 g. degreased leather are soaked in water overnight and then extd. with water at 45° at such rate as to give 1 l. of ext. in 3 hrs. The ext. is filtered through paper, the first 200 cc. being rejected, and 50 cc. are evapd. and then dried in a vacuum oven for 2 hrs. The English method gives results consistently higher on all samples tested. (Both methods give results much below the true values because of incomplete extn.—ABTRACTOR.) J. A. WILSON

A criticism of Baldracco and Camilla's modification of the official method. E. SCHRELL. *Le cuir* 9, 327–8(1920); *J. Soc. Leather Trades' Chem.* 4, 220–1(1920); cf.

C. A. 14, 474, 3544.—Absence of tannin in the non-tannin filtrate is not the only criterion of the value of a method of tannin analysis; the proposed method includes more non-tannin as tannin than is the case with the official method and it is neither well defined nor exact.

J. A. WILSON

Determination of bisulfites in tanning extracts. A. T. HOUGH. *Le cuir* 9, 162-3 (1920).—Dissolve 5 g. ext. in 100 cc. warm water, add 10 cc. satd. Br water, mix well, add 10 cc. 10% HCl, boil to expel excess Br, and ppt. sulfates as BaSO₄ in usual manner. Repeat, leaving out the Br water. The difference in wt. of BaSO₄ found in the 2 cases is a measure of the sulfite content of the ext.

J. A. WILSON

Determination of the permeability to water of sole leather. R. JALANE. *Le cuir* 9, 69-72, 296-300, 372-7 (1920).—An app. is described which consists of two nickel-plated copper plates, each 5 mm. thick and having a circular opening 36 mm. in diam. The lower plate holds 3 screws capable of swinging into grooves in the rim of the upper plate. The wide rim of a metal funnel is soldered to the upper plate while the narrow end is cemented to a glass tube of 6 mm. diam. graduated both as to height and vol. When a piece of leather 5 cm. sq. is placed between the two plates and the screws are swung into place and tightened, no water in the tube above can leak out except by passing through the 10 sq. cm. of leather exposed to the water. In making a test, the tube is filled with water to a height corresponding with the desired pressure; the rate of absorption of water is followed by means of the vol. graduations; and the time required for complete penetration is detd. by noting when water first appears on the under side of the leather, for which purpose it is convenient to place a mirror below the app. Contrary to the prevailing opinion, the grain layer of leather is not highly resistant to penetration by water. Leathers with greater densities are more resistant than lighter ones. Bleaching makes leather more permeable. A false resistance is produced by coating the grain with a drying oil and also by high water-sol. content; a leather containing 21.50% water-sol. matter required 24 hrs. for penetration, but when this matter was washed out and the leather was again dried, water would penetrate it in 30 mins. Leathers with a degree of tannage lying between 70 and 80 are more resistant than those more or less heavily tanned. Few commercial leathers are very resistant.

J. A. WILSON

Rapid estimation of chromium in chrome liquor by use of the immersion refractometer. ARTHUR W. THOMAS AND MARGARET W. KELLY. *J. Am. Leather Chem. Assoc.* 15, 665-8 (1920).—The authors have devised a rapid method for the detn. of the chrome content of chrome liquors. Detns. of the % are made with an immersion refractometer of the chrome soln. at 25°, containing not over 20 g. Cr₂O₃ per l. A table is given for converting % values to g. Cr₂O₃ per l. Temp. must be kept at 25° and the refractometer scale adjusted to read 13.25 for distd. water at 25°. High results are caused by the presence of impurities such as Al and Fe salts, NaCl, etc. The method has an accuracy of 0.1 g. Cr₂O₃ per l.

J. S. ROGERS

Some observations on the determination of the true tanning value of extracts according to Wilson and Kern. G. W. SCHULTZ AND T. BLACKADDER. *J. Am. Leather Chem. Assoc.* 15, 654-64 (1920).—S. and B., in the belief that some of the assumptions made by Wilson and Kern concerning their method for the detn. of tannin were fundamentally wrong, have investigated this method and reported the data obtained. The two assumptions which they consider in error are: (1) "that there is a definite chem. compd. formed between the tannin in the tannin soln. and the collagen in the hide powder during a 6-hrs. shaking, which chem. compd. is stable with regard to water and also insol. in water.; (2) that one definite chem. compd. is formed throughout the whole of the hide powder used in the analysis." Their results indicate that if hide powder is shaken in a tannin soln. of 2 or 3 times "analytical strength" for 6 hrs., the tannin is practically all removed from soln. But when the tanned hide powder is

treated with water by successive washings it appears that something is removed from the tanned hide powder which will in turn tan a second portion of hide powder, thus showing that although the tannin is completely removed from soln. by 6 hrs. shaking, it is not all permanently combined with the hide in that time. S. and B. further show that the finer particles of hide powder are more thoroughly tanned than are the coarser ones, and that these fine particles are easily lost when the tanned hide powder is squeezed through cloth, thus leaving the relatively tannin-poor hide powder behind for the subsequent analysis and detn. of tannin. They show that the wash waters obtained by the Wilson and Kern method when concd. under vacuum give a soln. containing tannin and which will tan hide powder. They point out that small errors in the detn. of N are magnified considerably in the tannin value obtained when tannin is calcd. according to Wilson and Kern method. The detailed analytical data are given in 7 tables. The results show that the method of Wilson and Kern gives low results due to the errors above mentioned.

J. S. ROGERS

Effect of concentration of chrome liquor upon the adsorption of its constituents by hide substance. ARTHUR W. THOMAS AND MARGARET W. KELLY. *J. Ind. Eng. Chem.* 13, 65-7(1921); cf. *C. A.* 14, 2563.—In accordance with the previous work of Miss Baldwin the max. absorption took place at a H^+ concn. of 0.00056 *M* per l. and a Cr_2O_3 concn. of 15 to 20 g. per l. Liquors varying in concn. from 0.363 g. to 202.03 g. Cr_2O_3 were used. It was found, as had been indicated by the work of Wilson and Gallun (cf. *C. A.* 14, 2728) that a minimum was reached, but contrary to their belief there was no subsequent increase in the amt. of Cr_2O_3 adsorbed. The minimum adsorption was reached when the liquor contained 147.5 g. Cr_2O_3 and this was maintained at 202 g. per l. At this highest concn. the liquor was so thick that a further increased concn. could not be handled. The authors believe that the evidence in this and previous papers indicates that the combination of hide substance and Cr_2O_3 is chem. and not physical as suggested by Davison (cf. *C. A.* 11, 1764).

I. D. GARARD

Use of formaldehyde in leather manufacture. JOSEPH JETTAMAR. *Le cuir* 9, 229-32, 242-6(1920).—A plumper leather may be obtained by swelling the hides with acid after they come from the stick vats and then fixing this swelling by treatment with formaldehyde. The tanning may then be completed in strong rocker liquors followed by drumming, but without the usual disadvantages. Formaldehyde may also be used for preserving hides, for fixing the hair of fur skins, and to stiffen the grain of loose hides. Tawed skins treated with formaldehyde, neutralized, washed, fat-liquored, and bleached are resistant to hot water.

J. A. WILSON

Theory of tanning dilute gelatin gels by formaldehyde. L. REINER. Frankfurt. *Kolloid-Z.* 27, 195-7(1920).—0.1 to 6% formaldehyde was added to 5% gelatin solns. By this treatment the softening temperature of the gelatin is raised. Moreover, contrary to the usual behavior of gelatin, the mixt. softens from the center outwards. R. explains this on the grounds that the tanning process does not occur homogeneously, but chiefly on the surface. The partially tanned gelatin has a greater attraction for the formaldehyde than the unaltered gelatin. Hence the reaction is to some degree auto-progressive. The process is reversible, for if the formaldehyde is driven off by the application of heat the original properties of the gelatin are restored. The presence of NH_3 retards the tanning action of the formaldehyde.

E. B. SPRAR

Nature of the insoluble in tanning extracts. A. T. HOUGH. *Le cuir* 9, 408-10, 439-41(1920).—Insol. matter in tan liquors is objectionable because it clogs the pores, necessitating scouring, and gives the grain a bad color. The insol. matter might be made available for tanning by peptization, but this action is very slow. Clear liquors actually produce better results. The amt. of insol. matter in exts. can be kept low by a careful peeling of the barks, extg. in open vats or in a vacuum rather than in autoclaves under pressure, by a suitable decantation of the exts., and by certain clarification methods.

Quebracho and similar exts. are rendered completely sol. by adding about 20% of certain synthetic tanning agents and heating to 50°, but not all synthetic materials are suitable. In some cases the free mineral acid must be neutralized before adding to the ext. Such mixts. penetrate and tan more rapidly, produce a clearer and finer grain, and make bleaching unnecessary.

J. A. WILSON

Tanning with silica. A. T. HOUGH. *Le cuir* 8, 209, 257, 314(1919).—Colloidal silicic acid resembles tannin in its property of pptg. gelatin from soln. Using an excess of silica, the ppt. obtained was found to contain 178.5 parts of silica per 100 of gelatin; with an excess of gelatin, only 109 parts of silica. An av. silica-tanned leather contains from 17 to 24% of SiO_2 . It is difficult to prevent a too great absorption of silica. A good tanning liquor is prepd. by pouring 30% Na_2SiO_3 into 30% HCl until the free acid is reduced to decinormal. This liquor will tan pickled sheepskins in 3 to 5 days or bull hides in a month. To make white leather, one has only to neutralize and fat-liquor. Attempts to combine vegetable and silica tannages were unsuccessful, probably because both tanning agents tend to combine with the same amino groups of the hide mol., but good leathers can be produced by a combined alum and silica tannage. If the stock is given a light chrome tannage first, the speed of the silica tannage is increased and a more solid leather results.

J. A. WILSON

Studies on technical casein. I. Introduction. W. MANSFIELD CLARK. *J. Ind. Eng. Chem.* 12, 1162-3(1920).—C. outlines the problem presented to the Dairy Division, Bureau of Animal Industry, to develop American-made casein in order to economize tonnage for the U. S. Shipping Board. II. Grain curd casein. W. M. CLARK, F. H. ZOLLER, A. O. KARLBERG AND A. C. WEIMAR. *Ibid* 1163-7.—When HCl is added to milk until methyl red indicates an apparent pH 4.6 (corresponding to pH 4.1 as detd. by the H electrode), the casein forms a resilient mass of small grainy curds, permitting extensive washing with water of like acidity without causing the usual objectionable packing. The technical method worked out is as follows: To sweet skim milk not over 90° F. is added a 1 to 8 diln. of 20° Bé. HCl until methyl red shows an apparent pH 4.8. Temp. is raised to 93-6° F., the acid being sprayed in with constant agitation. Wash with very dil. HCl (pH 4.6-4.8), drain, press, grind, and dry. Samples prepd. in the lab. showed 1.36 to 2.03% ash, factory-made samples 1.90 to 2.86, which is much below caseins prepd. by the cooked curd, H_2SO_4 , or natural sour processes. Grain-curd casein meets all the analytical requirements of the govt. specifications for use in airplane glue. It is low in moisture and responds rather slowly to atm. humidity. III. Methods of analysis. ROSCOE H. SHAW. *Ibid* 1168-71.—Moisture: for reliable results dry to const. wt. in partial vacuum at b. p. of water. Ash: char without permitting flame, then ignite in muffle (preferably elec.) at temp. just sufficient to yield white ash. Fat: modified form of Roesse-Gottlieb method. Sugar: modified form of method of Bryan, Given and Straughn (*C. A.* 5, 2875). P: Det. by official method for P in fertilizers (Bur. of Chem., *Bull.* 107, 1908). Ca: Det. in ash or by digestion of casein with aqua regia. In a note W. M. Clark states that by slightly overstepping the isoelectric point, grain-curd method tends to release the casein from basic combination, extensive washing removing ash-forming substances and leaving only a trace of acidity. IV. Standardization of the borax solubility test for commercial caseins. HARPER F. ZOLLER. *Ibid* 1171-3.—Fifteen g. casein ground to pass a 40-mesh sieve are vigorously stirred with 100 cc. of 0.2 M borax soln. at 30° in a 250 cc. beaker. Caseins prepd. by different methods show wide variations in consistency. See original for curves showing viscosities detd. by MacMichael viscosimeter, etc. J. A.

FRUDENBERG, KARL: Die Chemie der natürlichen Gerbstoffe. Berlin: Julius Springer. M. 22. For review see *Collegium* 1920, 431.

Curing hides or skins. G. R. OLLIPHANT. U. S. 1,362,502, Dec. 14. Hides are cured and tanned by submerging them in an aq. soln. containing constituents leached from acid iron earth.

Tanned leather belts. F. GILARDINI. U. S. 1,360,923, Nov. 30. Material for leather belts is tanned in successive sections while the material between the sections being tanned is subjected to compression to produce a product alternate small sections of which are untanned and of great strength.

30--RUBBER AND ALLIED SUBSTANCES

JOHN B. TUTTLE

Analysis of vulcanized rubber. MORISSON. *Caoutchouc & guttapercha* 17, 10233 (1920).—A procedure is given for the detn. of resins, fillers, free and total S, oil substitute and rubber, in samples of vulcanized rubber. FRANCES MCGOVERN

Rubber as a colloid. D. F. TWISS. *Caoutchouc & guttapercha* 17, 10240-3 (1920).—A review. FRANCES MCGOVERN

The properties of the colloidal state and their applications to industry. W. C. MCLEWIS. *Caoutchouc & guttapercha* 17, 10261-2 (1920).—A review. F. MCG.

The theory of vulcanization. GASPARI AND PORRITT. *Caoutchouc & guttapercha* 17, 10277-83 (1920).—A review. FRANCES MCGOVERN

Action of lipases on oils vulcanized with sulfur chloride (white substitutes). ANDRÉ DUBOSC AND JEAN WAVELET. *Bull. soc. ind. Rouen* 47, 47-59 (1919).—Oils, treated with S_2Cl_2 (the so-called *white substitutes*), gradually darken on standing. One sample, that darkened very rapidly, contained a considerable quantity of free fatty acid. Some samples of white neutral vulcanized oils were treated with a lipase soln. prepd. from castor-oil seeds and were heated in an oven at 35-40°. After several hours, the samples darkened rapidly; examn. showed free fatty acid and glycerol. It is, therefore, concluded that the darkening of white substitutes is caused by substances such as lipase, and that the oils should be sterilized if intended for the production of white substitutes. FRANK MCGOVERN

The action of heat and light on vulcanized rubber. JOHN B. TUTTLE. *The Rubber Age* 8, 271-2 (1921).—Vulcanization is the chem. reaction between rubber and S. At ordinary temps. the speed of reaction is practically zero and increases with rise in temp. Vulcanization is ordinarily effected between 275° and 300° F. but it is shown that the reaction has an appreciable speed at 212° F. even in the absence of catalysts. The action of heat on vulcanized rubber is to produce after- or over-vulcanization, with consequent deterioration, the effect being chiefly noticeable in the drop in the tensile properties, there being practically no change in the percentage of acetone-sol. matter. The action of sunlight is one of oxidation, the products of which are sol. in acetone. The acetone ext. curve for sunlight exposure tests, and for the natural aging of rubber, are very similar, from which it is concluded that the change in the natural aging is also one of oxidation. After oxidation has started, it proceeds with greatly increased speed, showing that small amts. of oxidized rubber act catalytically to oxidize the rest. Reclaimed rubber caused a break in the acetone ext. curve much sooner than in samples where it was not present. A further difference between the action of heat and sunlight is that the former acts throughout the entire mass, while the latter is a surface action only. J. B. T.

Hevea in Indo-China. I. General. AUG. CHEVALIER. Congrès d'agr. coloniale Série Saigon, *Bull.* 7, 1-18 (1918).—Most rubber plantations are in the eastern provinces of Tay Ninh, Thudaumot, Bienhoa, Baria and Giadinh, on what are known as the red and the gray soils. The latex is generally coagulated by $AcOH$ (1 g. per l. latex) pre-

viously dild. in H_2O . Some plantations use smoke. Magen has patented a process of coagulating the latex with the products collected in the distn. of wood, after the sepn. of AcOH and MeOH.

ALBERT R. MERZ

Hevea in Indo-China. V. Comparative coagulation of Hevea latex by sucrose, glucose and acetic acid. E. GIRARD AND E. ROSG. Congrès d'agr. coloniale, Sérié Saigon. *Bull.* 7, 45-8(1918).—Of 9 pure cultures of bacteria isolated from *Hevea* latex, 1 fermented glycerol and mannitol; 3, lactose; 4, sucrose; 5, maltose; and 7, glucose. To 3.5 l. lots of latex, there was added 1 l. 0.9% sucrose; 1 l. 0.85% glucose; 1 l. 0.9% sucrose acidified with AcOH; and 1 l. dil. AcOH, resp. Coagulation took place in 21 hrs. with the latex to which sugar was added; more rapidly with the acidified latex. The yields of dried gum were 0.300 kg., 0.250 kg., 0.250 kg. and 0.300 kg., resp. The serums from the sugared latex appeared more cloudy than from the acidified. The consts. detd. were:

	Sucrose added.	Glucose added.	Sucrose and AcOH added.	Acetic added.
Vol. of serum l.....	3.010	3.240	3.080	3.010
Acidity in AcOH per l.....	1.26 g.	1.20	1.86	1.20
Dry ext.....	7.08	6.40	6.54	6.00
Ash.....	1.84	0.92	1.44	1.60
Total N.....	0.31	0.31	0.31	0.36
Corresponding albuminoids....	2.13	2.04	2.04	2.15
Sucrose.....	0	0	0	0
Glucose or invert sugar.....	0	0	0	0

Glucose and sucrose may, therefore, replace AcOH for coagulating *Hevea* latex. The quantities of sugar used appear necessary and are sufficient. There is a difference in the coagulations by sugar and acid, and also in coagulation by weak and strong AcOH which will be explained in a future paper.

ALBERT R. MERZ

The testing of rubber. A. VAN ROSSEM. *Kolloidchem. Beihefte* 12, 103-73 (1920).—This is largely a review of the work of the Institute between 1912 and 1916, covering the investigations on methods for the phys. and chem. testing of rubber, a large part of which, in some form or another, has appeared in separate publications. (Cf. C. A., 12, 638, 2260; 13, 1547). For the evaluation of raw rubber, R. prefers to use physical tests made upon samples vulcanized from a mixt. of 92.5% rubber, 7.5% S. An oil bath is used instead of the usual steam vulcanizer on account of the difference between the sp. heat of the steam and oil, since with the latter the vulcanization temp. can be reached more quickly, and held more const., than can be done with steam. The degree of vulcanization is detd. by the coeff. of vulcanization, obtained as follows: 11 g. $Mg(NO_3)_2$, 30-40 cc. HNO_3 , 1.40 sp. gr. and 1 g. extd. rubber are placed in a round-bottomed flask, and after the first violent action is over, the flask is heated for 1.5 to 2 hrs. and the soln. evpd. to dryness. The HNO_3 is removed with HCl, the residue dissolved in water and HCl, and the sulfate in this soln. pptd. with $BaCl_2$ in the usual way. The free S is detd. by the method of Franck and Marckwald. The relative efficiencies of the various testing machines are discussed; the Schopper machine is given the preference over the other types because of the facility and accuracy with which stress-strain curves can be drawn. The permanent set is not considered of much value, being in a sense merely confirmatory of the results obtained from other tests, and containing such large errors that it is not as safe as the breaking tests made with the Schopper dynamometer. *Ibid* 174-231.—This is a discussion of the effect of various factors, such as washing, milling, temp. of vulcanization, percentage of S added, viscosity of the crude rubber, resins, ash, acidity and N in the crude rubber; the influence of fillers and accelerators; previous treatment of the rubber, etc. Mastication (milling) reduces the relative viscosity of the soln. of rubber in benzol,

whereas the coeff. of vulcanization remains constant. Increasing the time of cure or increasing the temp. raised the coeff. of vulcanization in a mixt. of 92.5 rubber and 7.5 S. Increasing the S (and consequently decreasing the rubber) likewise raised the vulcanization coeff. A table is given showing the effect on the coeff. of various inorg. fillers, neutral, basic and acid, as well as several accelerators. In rubber-S mixts. the viscosity of the raw rubber appears to have some effect on the vulcanization coeff., the latter increasing with increase in the viscosity number. However, with $1/8$, $1/4$, $1/2$ and $1/3\%$ *p*-nitrosodimethylaniline, it decreases with increase in the viscosity number.

J. B. T.

Lubrication in the rubber industry. ANON. *Lubrication* 6, No. 10, 1-9(1920).—General.

E. J. C.

The chemical examination of antimony sulfides (VAN ROSSEM, DEKKER) 7.

Bamber's Rubber Calculator Book. In the English and Metric Systems. London: Maclaren & Sons, Ltd. 1.50s. For review see *Gummi-Ztg.* 35, 271(1920).

FLEURY, R. DE: *Technologie du caoutchouc souple*. Paris: Dunod. 210 pp. 18 fr.

HANCOCK, THOMAS: *Personal Narrative of the Origin and Progress of the Caoutchouc or India-Rubber Manufacture in England*. Published by Longman, Brown, Green, Longmans and Roberts, London, 1857. Reprinted by order of James Lyne Hancock, Ltd. Vulcanized Rubber Works, 266 Goswell Rd., London, E. C. 72 pp. For review see *Chem. Weekblad* 17, 580(1920).

Rubber substitute. H. H. HAZELTINE and M. GREGORY. U. S. 1,360,744, Nov. 30. A rubber-like material is prepd. by heating a mixt. of S 10 and fish oil, 90%, or similar fixed oils free from air and moisture under pressure until reaction of the materials is completed. The product is similar to rubber in many properties and may be used with it.

Treating raw rubber. J. V. WORTHINGTON and A. W. T. HYDE. U. S. 1,360,486, Nov. 30. Raw rubber is heated in an atm. of non-oxidizing gas such as live steam free from air in order to produce a product of uniform softness, which is then dried *in vacuo*. The steam treatment may last 3-7 hrs. at 10-60 lbs. pressure per sq. in.

Rubber sponges. G. W. BELDAM. Brit. 151,084, June 14, 1919. Rubber sponges are formed so as to have large and small pores in different parts of the sponge, either by vulcanizing together superposed layers of two different compds. which will yield on vulcanization portions having large and small pores, or layers of the same compd. 1 part of which has been masticated more than the other and forms larger pores on vulcanization. Alternatively, a mixt. of the two compds. is vulcanized while supported in a shallow vessel of H₂O with its lower part dipping into or just about the H₂O. A compd. which yields on vulcanization small pores consists of Para rubber, milk of S, lithopone, crimson sulfide of Sb or vermilion, ceresin wax, pine oil, together with (NH₄)₂CO₃ or amyl acetate. In a compd. for yielding large pores, larger proportions of carbonate or amyl acetate are used, pptd. chalk and ZnO are used in place of lithopone and turpentine may partially replace pine oil.

